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U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN   No. 38.

PROCEEDINGS

OF THE

TENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

CHICAGO, ILLINOIS,

AUGUST 24, 25, AND 26, 1893.

EDITED BY

HARVEY W. WILEY,

SECRETARY OF THE ASSOCIATION.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

NOTE.

In the spelling of chemical terms this bulletin conforms in the following particulars to the report made to the American Association for the Advancement of Science, at its fortieth meeting by the committee on spelling and pronunciation (see proceedings of the association for 1891, pp. 175, 176).

Names of chemical substances having the termination—**ide** drop the final **e**, as chlo′rid, i′odid, hy′drid, o′xid; and those having the termination—**ine** drop the final **e**, as chlo′rin, bro′min.

The termination—**ol** is used exclusively for alcohols, as glycerol, not glycerine.

II

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., September 13, 1893.

Sir: I beg to submit herewith, for your inspection and approval, the manuscript of the proceedings of the tenth annual convention of the Association of Official Agricultural Chemists, at Chicago, Ill., August 24, 25, and 26, 1893.

Respectfully,

H. W. Wiley,
Chief of Division of Chemistry and Secretary of Association of Official Agricultural Chemists.

Hon. J. Sterling Morton,
Secretary of Agriculture.

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FIRST DAY.

THURSDAY.

In accordance with the call of the executive committee the association met in the Memorial Art Palace, Chicago, Ill., at 1 o'clock p. m., the president, Mr. S. M. Babcock, in the chair.

The following members, and others interested in the objects of the association, were present:

MEMBERS AND VISITORS PRESENT.

The president, Mr. S. M. Babcock, Chemist and Professor of Agricultural Chemistry, University of Wisconsin, Madison, Wis.

The secretary, Mr. H. W. Wiley, Chief of Division of Chemistry, U. S. Department of Agriculture, Washington, D. C.

Of the executive committee, Mr. B. W. Kilgore, Chemist, North Carolina Experiment Station, Raleigh, N. C.; and the following members and participants:

Atwater, W. O., Professor of Chemistry, Wesleyan University, and Director Storrs Experiment Station, Middletown, Conn.

Bigelow, W. D., Assistant in Chemical Laboratory, U. S. Department of Agriculture, Washington, D. C.

Blouin, R. E., Assistant Chemist, Louisiana Experiment Station, Baton Rouge, La.

Chazal, P. E., Chemist, Shepard Laboratory, Charleston, S. C.

Cochran, C. B., Inspector of Foods, Pennsylvania State Board of Agriculture, West Chester, Pa.

Collingwood, C. B., Chemist, State Agricultural Experiment Station, Tucson, Ariz.

Crampton, C. A., Chemist, Bureau of Internal Revenue, Washington, D. C.

Curtis, H. E., Chemist, Kentucky Experiment Station, Lexington, Ky.

De Roode, R. J. J., Chemist, West Virginia Agricultural Experiment Station, Morgantown, W. Va.

Doremus, C. A., Adjunct Professor, Bellevue Hospital Medical College, New York City.

Eaton, C. N., Chemist, Minnesota State Dairy and Food Commission, St. Paul, Minn.

Ewell, E. E., Assistant in Chemical Laboratory, U. S. Department of Agriculture, Washington, D. C.

Farrington, E. H., Chemist, Experiment Station, University of Illinois, Champaign, Ill.

Frear, W., Chemist, State Board of Agriculture, Agricultural Experiment Station, Pennsylvania State College, State College, Pa.

Fries, J. A., Assistant in Chemical Laboratory, Agricultural Experiment Station, State College, Pa.

Gaines, R. H., Chemist, Virginia Department of Agriculture, Richmond, Va.

Hartwell, B. L., Assistant Chemist, Rhode Island Agricultural Experiment Station, Kingston, R. I.

Huston, H. A., State Chemist of Indiana and Professor of Agricultural Chemistry, Purdue University, Lafayette, Ind.

Kebler, L. F., Analytical and Manufacturing Chemist, Smith, Kline & French . Company, Philadelphia, Pa.

Kedzie, R. C., Professor of Chemistry, Agricultural Experiment Station, Agricultural College, Mich.

Lindsey, J. B., Amherst, Mass.

Lord, N. W., Professor Mining, Metallurgy and Chemist for State Board of Agriculture, Ohio State University, Columbus, Ohio.

McDonnell, H. B., Professor of Chemistry, Maryland Agricultural College, College Park, Md.

McElroy, K. P., Assistant Chemist, U. S. Department of Agriculture, Washington, D. C.

Mitchell, G. A., Wesleyan University, Middletown, Conn.

Parsons, C. L., Professor of General and Analytical Chemistry, New Hampshire College, Hanover, N. H.

Payne, G. F., State Chemist, Georgia Agricultural Department, Atlanta, Ga.

Perkins, W. R., Agricultural College, Miss.

Rawls, W. A., State Chemist of Florida, Tallahassee, Fla.

Rising, W. B., State Analyst for California, State Board of Health; Professor of Chemistry, University of California, Berkeley, Cal.

Ross, B. B., Professor of Chemistry, Louisiana State University and A. and M. College, and Chemist, State Experiment Station, Baton Rouge, La.

Schweinitz, E. A. de, Chemist, Bureau of Animal Industry, U. S. Department of Agriculture, Washington, D. C.

Scovell, M. A., Director Kentucky Agricultural Experiment Station, Lexington, Ky.

Sederholm, E., Assistant Professor, Technical College of Stockholm, Stockholm, Sweden.

Sharples, S. P., Assayer and Inspector of Liquors, Commonwealth of Massachusetts, Boston, Mass.

Sherman, H. C., Assistant in Chemistry, Maryland Agricultural College, College Park, Md.

Shutt, F. T., Chemist, Experimental Farm, Ottawa, Canada.

Slosson, E. E., Chemist, Wyoming Agricultural Experiment Station, University of Wyoming, Laramie, Wyo.

Smith, H. M., State Station, New Haven, Conn.

Street, J. P., Assistant Chemist, New Jersey State Experiment Station, New Brunswick, N. J.

Stubbs, W. C., State Chemist, Louisiana State Experiment Station, Audubon Park, New Orleans, La.

Teller, G. L., Chemist, Arkansas Agricultural Experiment Station, Fayetteville, Ark.

Trescot, T. C., Assistant in Chemical Laboratory, U. S. Department of Agriculture, Washington, D. C.

Trubek, M., Chemical Engineer, William Zahn Leather Company, Newark, N. J.

Van Slyke, L. L., Chemist, New York Agricultural Experiment Station, Geneva, N. Y.

Voorhees, L. A., Chemist, New Jersey Agricultural Experiment Station, New Brunswick, N. J.

Wahl, R., Chemist, Wahl & Henius, Chicago, Ill.

Winton, A. L., Chemist, Connecticut Agricultural Experiment Station, New Haven, Conn.

Woll, F. W., Assistant Chemist, Wisconsin Agricultural Experiment Station, Madison, Wis.

On motion, Mr. Huston was appointed by the president to act as secretary *pro tempore.*

A motion that the president appoint a committee of three on order of business was carried, and the president appointed as such committee Messrs. Huston, Van Slyke, and Woll.

A recess was taken until 2 o'clock.

Upon reassembling, Mr. Huston, for the committee on order of business, reported the following

PROGRAMME.

1. President's address.
2. Report on phosphoric acid.
3. Report on potash.
4. Report on nitrogen.
5. Report on soils and ash.
6. Report on dairy products.
7. Report on cattle foods.
8. Report on fermented liquors.
9. Report on sugar.
10. Report of abstract committee.

The programme as proposed was adopted.

The president read his address, as follows:

ADDRESS OF PRESIDENT S. M. BABCOCK.

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: It gives me great pleasure to welcome you to the tenth annual meeting of this association. Since its organization upon the present basis, which was consummated at Philadelphia in 1884, this is the first time that the meeting has not been called at Washington. An exception has been made this year by the executive committee, only because it was believed that the World's Fair and the scientific congresses connected with it would attract to this place, at this time, a larger and more representative body of men interested in our special line of work than could be secured at the usual place. Especially was it hoped that representatives of foreign societies of agricultural chemists might meet with us here and perhaps take part in our discussions. I trust that we may not be disappointed in this, as the World's Fair has done much to reduce the data presented for discussion by the several reporters. Unfortunately, so far as this meeting is concerned, many of our most active members have been obliged to give much of their time during the past year, either to the preparation or care of exhibits, and in consequence have been unable to do the work asked by the reporters. I trust, however, now that we are here, that the many attractions which the fair and this great city offer may not prove

stronger than the work in hand, and that the members will attend to the business which has called them together before giving much time to pleasure. From what I have been able to learn I believe that but few changes in method will be recommended by the reporters. If this is true we can, by confining the discussion to the topics under consideration, make the meeting a short one.

The primary object of this association, as defined by its constitution, is to secure uniformity and accuracy of the methods, results, and modes of statement of analyses of the materials connected with the agricultural industry. The methods heretofore recommended have been very generally adopted by agricultural chemists in this country, and, in consequence, have contributed greatly to the value of results in agricultural experiment stations, as their work may now be compared directly—a thing impossible when the methods used were widely different.

The active members of this association are connected either with the Department of Agriculture at Washington, with the agricultural experiment stations, or with the agricultural colleges. All of them are, therefore, deeply interested in the promotion of agriculture, and most of them are directly engaged in investigations designed to throw more light upon some of the varied problems which confront the practical agriculturist. Taking into consideration the class of men which makes up this body, it would be expected that the methods recommended here would be well adapted to the solution of such problems and entirely consistent with the varied needs of agriculture. In general, I believe this is the case. The methods of fertilizer analysis are well adapted to show the amount and relative value of the constituents of plant food which different fertilizers are supposed to contain. The methods for the analysis of dairy products are well adapted to secure the ends desired, and the same may be said of the technical methods for the examination of sugar and for the analysis of fermented liquors. There is, however, in my opinion, a notable exception to this rule in the methods generally used for fodder analysis—a line of work which, next to the examination of commercial fertilizers, appropriates more of the time of agricultural chemists than any other line. It seems strange, under such circumstances, that these methods, designed to show the true value of fodders, should fail to determine a single constituent which, from the results of the analysis alone, can be classed as a food, if we except the water which the fodder contains. The present system, therefore, is utterly unable to discriminate between a first-class fodder and a substance totally unfit for food. As an illustration of what I mean I may mention the fact that the dried excrements, analyzed in digestion experiments, often give figures which correspond almost exactly with those from some of the best feeds. It is also possible, by using in proper proportions such substances as leather scraps, sawdust, oxalic acid, and kerosene oil, to prepare a mixture which, when analyzed by the official method, will give results which correspond with

those from any fodder. It seems to me that methods which are capable of such an interpretation are not in harmony with the progressive spirit of this association, and I believe the time has come when we should take active measures to replace it with something better.

If, now, I am asked what can be done to throw more light upon the composition and value of fodders than is done by the present method, I would suggest a closer study of the nitrogenous principles of fodders, and that an attempt be made to identify some of the constituents which make up the nitrogen-free extract of the present system. I would determine the sugar, the starch, and the gums; and, so far as practicable, I would discriminate between the pure cellulose, the lignins, and other constituents of crude fiber. I would separate the glycerides from the ethereal oils, the coloring matter, and the wax, which the ether extract contains. In general, I would substitute for the present illogical, indefinite, and unmeaning system, one which, so far as it went, would deal with the proximate constituents which the fodders contain.

I am aware that the scheme which I have outlined would require much more time than the present, and that the number of analyses turned out each year would be very greatly reduced by its adoption. But is it not better, is it not more to the credit of this association and the experiment stations which it so largely represents, to be able to point to a few facts which have the same meaning yesterday, to-day, and to-morrow, and which can be applied to all fodders, whatever their source, than to collect a great mass of unmeaning generalities, such as our present system supplies, and which no one except the agricultural writer who, for so much a column, manufactures rations on demand to fit all sorts of conditions, is able to interpret?

Progress along the lines suggested need not be slow, for we have a large number of workers; and if these will devote one-half the time to this subject which is now wasted in endeavors to make the sum of analytical errors represented by the term nitrogen-free extract agree in duplicate analyses to the second decimal place, we will in the next decade not only have methods which are applicable to this work, but we will have collected a mass of facts which can not fail to throw new light upon feeding problems.

Nor will the practical feeder suffer any inconvenience, even if analysis by the present system cease altogether, for at best he can use only average figures, it being impracticable to have analyses made of each lot of food obtained, and these figures are now so numerous for all of the standard feeds that the average is not changed one-tenth of 1 per cent by the hundreds of analyses reported each year.

I hope, therefore, that the reporters on fodder analyses appointed for the coming year will try to have some systematic work done along the line suggested. It should not be left to individual effort, as at present. We need concerted action.

The president, at the conclusion of his address, called on Mr. De Roode for the report on phosphoric acid.

REPORT ON PHOSPHORIC ACID.

By R. J. J. De Roode.

As early as possible after receiving formal notification of my appointment as reporter on phosphoric acid, I prepared circular letters which were mailed to over two hundred chemists and manufacturers of fertilizers. The object of these letters was to ascertain the opinions of as many competent judges as possible as to the defects in our present official methods for determining the different forms of phosphoric acid in fertilizing materials, and also to obtain suggestions as to the best means of remedying these defects.

Numerous replies were received, and all criticisms and suggestions were tabulated. As a result, I found that it would be entirely out of the question to take up for investigation all of the subjects which were suggested, and it became necessary to select for this year's investigation those subjects which were requested by the largest number, and to arrange these subjects in the order of importance. This was done and the subjects selected in their order of importance were as follows: (1) Citrate-insoluble phosphoric acid; (2) Determination of iron and alumina in phosphates; (3) The Kjeldahl methods for total phosphoric acid; (4) Determination of moisture; (5) Soluble phosphoric acid. Two samples were prepared—No. 1, a mixed fertilizer, and No. 2, a finely ground native rock phosphate from Florida.

Sample No. 1 was prepared by mixing about 150 samples of various brands of fertilizers sold in the State of West Virginia. No samples of raw bone were added. All other samples, mixed fertilizers, special fertilizers, acid phosphates, natural guanos, etc., were mixed together and sifted through a 40-mesh sieve. The coarse portion was discarded, and the fine portion spread out in a warm dry place and thoroughly air dried. It was then thoroughly mixed by sifting a large number of times through a coarse sieve. The fertilizer thus prepared was divided into 60 squares of equal size. Sixty bottles were placed in a row and with a small scoop, shaped like a thimble, the same quantity from the first square was added to each of the sixty bottles. Then the same quantity from the second square was added to each bottle, then from the third square, and so on, until the same quantity from each of the 60 squares had been added to each of the 60 bottles. I feel confident that there can be no possible variation in the composition of the samples, and, as they contain representatives of almost every kind of fertilizing material on the market except raw bone, any method of analysis which will give good results upon this sample ought to give good results upon any fertilizer.

Sample No. 2 was furnished to me by Mr. N. Robinson, of Florida. It was finely ground and passed through an 80-mesh sieve. It was then thoroughly mixed, divided into squares, and the bottles filled, as in the case of sample No. 1.

The bottles were all stoppered at the same time by tightly fitting rubber stoppers, thereby rendering any change from loss or gain of moisture impossible. The contents of the bottles were then thoroughly mixed by shaking and rolling. Very explicit and concise printed instructions were prepared, setting forth exactly what was desired, and copies of these instructions were sent with each set of samples. In reply to circular letters requesting coöperation in this work, I received letters from fifty-one chemists who expressed their desire or willingness to do work. Samples and instructions were mailed to each of these fifty-one chemists, and their receipt in good condition was acknowledged. I regret very much, however, that results of work upon the samples were received from but a very small percentage of these fifty-one chemists, and that some of these results were quite discouraging. A few chemists have followed the instructions and have submitted most excellent reports. Others have submitted only partial reports upon one or two scattered subjects. Still others have evidently either not read the instructions at all, or have paid no attention to them, and have merely put the samples through a regular fertilizer

analysis by their own laboratory methods and reported results without any description of the details of the methods used, just as if they were analyzing a sample of fertilizer for a manufacturer and reporting the results to him.

It is thus evident that quite a large amount of time has been wasted in useless work, which, if it had been properly spent and the instructions followed, might have led to valuable results. Some results have been received which were evidently incorrect and were omitted altogether in this report. These, however, were few in number. A number of chemists who are not members of this association have expressed their desire or willingness to lend their aid, and were furnished with samples and instructions. Some of these have not been heard from. A few of them have reported, however, and their reports are among the best which I have received. In my opinion, this association should encourage the coöperation of all chemists who are not its members, especially in view of the fact that its own members show such a remarkable lack of interest in the coöperative work.

The first subject called for in my instructions was that of the determination of the citrate-insoluble phosphoric acid in sample No. 1. In almost every communication which I received in reply to my circular letters, particular mention was made of the unsatisfactory results obtained by different chemists using our present official method for this determination. Now there is no doubt in my mind as to the chief cause of the discrepancies among chemists in their results on citrate-insoluble phosphoric acid, and this cause is to be found in the fact that their working conditions are different. There may be slight differences in the neutrality of their citrate solutions. We all know that it is very hard, if not impossible, to prepare strictly neutral ammonium citrate solution. But errors arising from this source are comparatively slight when we consider the differences which are introduced by the other varying conditions which are admitted by trying to follow our present directions for the "thirty minutes digestion at 65° C."

It occurred to me that almost all of these different conditions might be eliminated by simply heating the 100 cc. of citrate to 65° C. before adding the fertilizer to it. If different chemists have 100 cc. of citrate standing at 65° C. and introduce 2 grams of a fertilizer into it and allow it to remain there for thirty minutes, it makes no difference what kind of apparatus is used, the conditions are necessarily the same in every case. A chemist may use his own judgment about the kind of flasks and water bath he will use, and may employ any devices he thinks desirable; all that is necessary is that he have such apparatus as will heat 100 cc. of citrate to 65° C. and afterwards keep it at exactly this temperature for thirty minutes. It will make no difference how long or how short a time it takes to raise the citrate to 65° C. as the fertilizer will not be introduced until it does reach 65° C. In accordance with this idea, I requested determinations of the citrate-insoluble phosphoric acid in sample No. 1, by the following method:

Heat 100 cc. of citrate solution to exactly 65° C. in a water bath as described, and subsequently maintain this temperature for half an hour. So soon as the citrate solution has reached 65° C. introduce the filter containing the washed residue into the flask and stopper tightly with a clean, solid rubber stopper without removing the flask from the bath. Immediately after stoppering remove flask and shake violently. Put it back into the bath and allow to remain for exactly thirty minutes from the minute of stoppering, removing and shaking violently every five minutes. Filter, using a pump and sucking off all the liquid. Now wash the flask and the substance on the filter with water of exactly 65° C. It is well to have two wash bottles standing in the water bath. They will be at exactly 65° C. and when one has cooled somewhat from use it is replaced in the bath and the other used, and so on till all the washings are completed. After thorough washing, suck as dry as possible, remove filter, burn off filters and all organic matter, and dissolve in strong hydrochloric acid. When solution is complete, dilute, heat, filter, and wash with hot water. Collect filtrate and washings in an appropriate vessel and precipitate

the phosphoric acid from the entire solution after neutralization and addition of ammonium nitrate in the customary manner, and proceed as usual. The object of precipitating from the entire solution is to give a more convenient sized precipitate to work with and also to diminish errors arising from dilution and taking different sized portions, some taking a portion corresponding to a half gram and some one gram. Please follow these directions minutely, and make duplicate or triplicate determinations. Also determine the citrate-insoluble phosphoric acid, using the method as ordinarily carried out in your laboratory, and report details of your method as minutely as possible. The results received are tabulated in the first column of Table I. The results in the second column were obtained by the various analysts using the old method as ordinarily carried out in their respective laboratories.

TABLE I.—*Citrate-insoluble phosphoric acid.*

Name of analyst.	New method.	Old method.
	Per cent.	*Per cent₃*
B. L. Murray, Geneva, N. Y	1.37	1.03
	1.33	1.08
	1.35	1.03
	1.33	....
F. B. Carpenter. Raleigh, N. C	1.35	1.62
	1.40	1.65
H. B. McDonnell, College Park, Md	1.50	1.66
	1.54	1.62
F. S. Shiver, Fort Hill, S. C	1.40	1.47
N. Robinson, Tallahassee. Fla	1.40	....
H. E. Harrison, Agricultural College, Michigan	1.46	....
	1.50	....
	1.54	....
K. P. McElroy, Washington, D. C	1.53	....
	1.58	....
J. A. Fries, State College, Pennsylvania	1.69	1.87
	1.66	1.86
Miss Jessie Dent, Morgantown, W. Va	1.45	1.62
R. De Roode, Morgantown, W Va	1.46	1.46
	1.45	1.50
	1.46	1.52
	1.43	1.54
	1.47	1.48
J. R. Hanahan, Charleston, S. C	1.42	1.39
Lehmann & Glaser, Baltimore, Md	1.46	....
A. W. Pratt, Atlanta, Ga	1.50	1.61
	1.41	1.74
S. T. Donaldson, Charleston, S. C	1.56	2.09
	1.55	2.11
G. W. Cavanaugh, Ithaca, N. Y	....	1.65
	....	1.60
R. E. Noble. Raleigh, N. C	....	1.51
	....	1.56
H. E. Curtis. Lexington, Ky	1.54	1.79
	1.57	1.89

It is thus readily seen that the results by the new method of treatment are as a rule more concordant and slightly lower than by the old method. I am quite confident that this method will give generally more concordant and more satisfactory results than the method as at present carried out. One can see at once that the

proposed alteration eliminates all of the sources of the discrepancies now so commonly met with. I do not claim that this alteration of the old process gives results which are any nearer the truth as regards the absolute availability of a phosphate. All that is claimed for it is that it introduces uniformity—a thing which is not attainable under the present wording of our method—and at the same time does not interfere with accuracy. Should a somewhat smaller percentage of insoluble phosphoric acid be obtained by its use, who will say that it is or is not nearer the truth. As regards true availability, we have no means of deciding upon the correct amount. The most we can hope for is to decide upon a method to be used by all chemists. Now, as such a method should above all things give concordant results in the hands of different chemists, I would strongly recommend that the alteration proposed be adopted. We need more uniformity in our work on citrate-insoluble phosphoric acid. The proposed alteration will attain this end, if not wholly, at least in part. It is something of an advance in the direction of uniformity, and it should not be overlooked.

Among the objections which might be urged against this method of procedure, the only one worthy of any consideration is this: That some change in the specific gravity and the neutrality of the ammonium-citrate solution might take place during the preliminary heating in an open flask. If the flask should be kept stoppered during the preliminary heating, and opened only just as the substance was introduced, no change, of course, would be possible. As it would be somewhat more convenient, however, to work with the flask open until the substance was introduced, especially in order to avoid the internal pressure which sometimes bursts the flask, I requested that 100 cc. of the ammonium citrate be heated to 65° C. in an open flask and then cooled and the specific gravity and neutrality tested. The results were as follows:

TABLE II.—*Ammonium-citrate solution.*

Name of analyst.	Sp. g.	Neutral or acid.
J. R. Hanahan, Charleston, S. C	1.0900	Neutral.
B. L. Murray, Geneva, N. Y	1.0900	Do.
H. B. McDonnell, College Park, Md	1.0900	Do.
H. E. Harrison, Agricultural College, Michigan	1.0900	Do.
H. E. Curtis, Lexington, Ky	1.0902	Do.
R. De Roode, Morgantown, W. Va	1.0902	Do.
S. T. Donaldson, Charleston, S. C	1.0905	Do.
J. A. Fries, State College, Pennsylvania	1.0906	Do.
F. S. Shiver, Fort Hill, S. C	1.0909	Do.
F. B. Carpenter, Raleigh, N. C	1.0918	Do.
Lehmann & Glaser, Baltimore, Md	1.0950	Acid.
Do	1.1000	Do.

Lehmann & Glaser report that after heating, the citrate showed a very slight acid reaction. They also report a considerable change in specific gravity. No other analysts found any change in the neutrality. Mr. Carpenter finds a decided change in specific gravity. The other analysts find either no change at all or only a very slight change. In some of the experiments the citrate was heated for a considerable time and in a beaker instead of a flask, so that these tests were hardly correct. Now, although I feel confident that the very slight change which takes place is too small to be of influence upon the result of a determination, I would, nevertheless, to be on the safe side, recommend that during the preliminary heating the flask be kept loosely stoppered. This will prevent evaporation and at the same time will produce no pressure within the flask.

The second subject which was taken up for investigation was the determination of iron and alumina in phosphates. Now, although I was aware of the fact that this subject did not come under the duties of the reporter on phosphoric acid, I felt that, owing to the numerous requests for investigation on this subject, something ought to be done. Sample No. 2 was prepared especially for this purpose, and the results of the determinations of iron and alumina in this sample are as follows:

TABLE III.—*Oxid of iron and alumina.*

Name of analyst.	Fe_2O_3.	Al_2O_3.	Fe_2O_3+Al_2O_3.
	Per cent.	Per cent.	Per cent.
Lehmann & Glaser, Baltimore, Md	2.28	1.80	....
	2.23	1.80	....
	2.17	1.89	4.10
	....	1.90	....
	....	1.92	....
F. S. Shiver, Fort Hill, S. C	1.19	1.85	3.06
	1.19	1.89	....
N. Robinson, Tallahassee, Fla	2.19	2.54	4.73
J. A. Fries, State College, Pa	1.34	.50	1.84
R. De Roode, Morgantown, W. Va	2.07	1.94	4.04
	2.16	1.91	....
J. R. Hanahan, Charleston, S. C	....	....	5.25
	....	....	5.10
	....	....	5.35
	....	....	5.30
W. D. Bigelow, Washington, D. C	....	....	5.53
	....	....	5.59
F. B. Carpenter, Raleigh, N. C	....	....	4.16
R. E. Noble, Raleigh, N. C	....	....	4.00
H. E. Curtis, Lexington, Ky	....	....	4.04
	....	....	4.00

These results admit of no conclusion as regards the best method. There are too few results obtained by any one method, and there is nothing to show which method gives results which are nearest the truth. In my opinion, the method of Mr. Charles Glaser, of the firm Lehmann & Glaser, of Baltimore, is a good one. This method is described by Mr. Glaser in the Zeitschrift für analytische Chemie **31**, 4, and the separation of the iron and alumina by fusion with alkali carbonate is described by him in the October, 1892, number of the Pharmaceutical Review, **1**, 10, published in Baltimore, Md.

It is the opinion of some of the members of this association that the determination of iron and alumina in phosphates is without the province of the investigations of this association. I, however, am of a different opinion, and, since this is a subject upon which the greatest controversies have arisen among chemists, I deem it the duty of the Association of Official Agricultural Chemists to take up the matter for exhaustive investigation, and to establish, if possible, a reliable method for the estimation of iron and alumina in phosphates, to be considered as an official method in the same light in which our other methods are official, and I would recommend that a special reporter be appointed next year for this purpose.

The third subject upon which work was requested in the instructions was the determination of the total phosphoric acid in samples No. 1 and No. 2 by the methods of digestion with sulphuric acid and such other reagents as are used in the Kjeldahl process and its several modifications for the estimation of nitrogen. It was believed that any one of these processes would give a solution in which the total phosphoric

acid could be estimated with accuracy, thereby rendering one weighed quantity of fertilizer and one digestion sufficient, a solution being thus obtained in portions of which both the nitrogen and the total phosphoric acid could be determined.

So far as the plain Kjeldahl method of digestion with sulphuric acid and mercury or mercuric oxid is concerned, it has been proved to give most excellent results and is now extensively used. It has also been fairly well established that the various modifications of the Kjeldahl process of digestion also furnish solutions in which the phosphoric acid may be accurately estimated. Some additional work seemed to be necessary on this point, however, and the results reported are given in Table IV.

TABLE IV.—*Kjeldahl methods.*

Name of analyst.	Modified Kjeldahl.	Plain Gunning.	Modified Gunning.
	Per cent.	*Per cent.*	*Per cent.*
A. W. Pratt, Atlanta, Ga..	13.34	13.20	....
	13.36	13.34	....
F. S. Shiver, Fort Hill, S. C..............................	13.08	....	....
	13.11	....	....
Miss Jessie Dent, Morgantown, W. Va...............................	13.13	13.16	....
	13.13	13.19	....
R. De Roode, Morgantown, W. Va......................................	13.09	13.10	13.07
	13.12	13.04	13.16
	13.04	13.16	13.10
	13.15	13.01	13.13
	13.11	13.20	13.21
B. L. Murray, Geneva, N. Y..	....	13.15	13.23
	....	13.05	13.20
	....	13.09	13.24
H. E. Curtis, Lexington, Ky...	13.32	13.38	....
	13.32	13.34	....

Sample No. 2.

Miss Jessie Dent, Morgantown, W. Va...............................	36.18	36.10	....
	36.26	36.16	....
R. De Roode, Morgantown, W. Va	36.30	36.22	....
	36.22	36.14	....
	36.36	36.30	....
	36.26	36.20	....
	36.16	36.32	....
B. L. Murray, Geneva, N. Y	....	36.36	36.77
	....	36.36	86.70
	....	36.46	36.82
H. E. Curtis, Lexington, Ky..	36.48	36.46	....
	36.52	36.58	....

Although the results are not so numerous as they might have been on all of these processes, they nevertheless seem to indicate quite clearly that any one of these modifications gives a good solution for the estimate of phosphoric acid, and I would recommend their use whenever the nitrogen and the total phosphoric acid are both to be determined in a sample of fertilizer. In order to compare these results with the results of determinations of total phosphoric acid by other methods, I have prepared Table V, which contains these results:

TABLE V.—*Total phosphoric acid by methods other than Kjeldahl.*

Name of analyst.	No. 1.	No. 2.	Method.
	Per cent.	*Per cent.*	
G. W. Cavenaugh, Ithaca, N. Y....................	12.56	....	
	12.66	....	
	12.75	....	
	12.76	....	"Official methods" is
	12.76	....	all that was stated.
	12.32	....	
	13.17	....	
	13.28	....	
N. Robinson, Tallahassee, Fla....................	12.70	36.06	HNO_3+HCl.
Lehmann & Glaser, Baltimore, Md................	12.98	....	$HCl+HNO_3$.
K. P. McElroy, Washington, D. C................	12.98	35.90	Ignition and solution
	13.04	35.86	in HCl.
F. B Carpenter, Raleigh, N. C..................	13.04	36.18	HNO_3+HCl.
	13.10	36.18	Do.
B. L. Murray, Geneva, N. Y....................	13.08	36.35	$HCl+KClO_3$.
	13.10	36.32	Do.
F. S. Shiver, Fort Hill, S. C..................	13.09	35.61	HNO_3+HCl.
R. E. Noble, Raleigh, N. C....................	13.10	36.20	HNO_3+HCl.
F. S. Shiver, Fort Hill, S. C..................	13.11	....	Burney.
S. T. Donaldson, Charleston, S. C............	13.18	....	Do.
	13.18	....	Do.
	13.18	....	HNO_3+HCl.
	13.24	....	Do.
A. W. Pratt, Atlanta, Ga......................	13.28	....	HNO_3+HCl.
	13.36	....	$Mg(NO_3)_2$.
J. A. Fries, State College, Pa................	13.38	36.79	HNO_3+HCl.
	13.39	36.87	Do.
J. R. Hanahan, Charleston, S. C...............	13.52	36.69	$HNO_3+HCl+KClO_3$.
	13.53	36.70	
H. E. Curtis, Lexington, Ky...................	....	36.52	Dissolved in HCl.
	....	36.44	

It is at once seen that there are larger discrepancies in these results than in the results by the Kjeldahl methods. These results are not, on the whole, so good as they should be and leave much room for criticism, either of the analysts or of the methods. I am inclined to think that the fault lies chiefly in the analysts themselves, as our methods have been proved to be good.

The fourth subject upon which I was requested to institute an investigation was the determination of moisture. I must admit that this is an important subject, but that it is a subject upon which about all has been done that is possible. After much consideration, I could think of nothing that would be of any improvement upon our present method, which is the result of much painstaking investigation. It is manifestly impossible to compel chemists to adopt exactly the same kind of drying oven, and as small differences in temperature have been shown to have large influences upon results, I can not see much prospect for further improvement in this direction. In my instructions upon this subject, I described the method a little more in detail than is usual, and requested moisture determinations by the following method:

Have a double-walled water oven provided with a constant level water supply. Heat the water till it boils briskly and then introduce the weighed 2 grams of sample No. 1 on a watch glass and allow it to remain for five hours, the water boiling briskly all the time and the constant level maintained. Remove, cover with a matched water glass, cool in dessicator and weigh. Results obtained were as follows:

TABLE VI.—*Moisture.*

Name of analyst.	Moisture.	Name of analyst.	Moisture.
	Per cent.		*Per cent.*
F. S. Shiver, Fort Hill. S. C...........	2.23	J. A. Fries, State College, Pennsylvania.	2.74
Lehman & Glaser, Baltimore, Md.....	2.49		2.75
R. De Roode, Morgantown, W. Va....	2.50	A. W. Pratt, Atlanta, Ga.............	2.89
	2.49		2.91
	2.54	J. R. Hanahan, Charleston, S. C......	3.32
	2.60	K. P. McElroy, Washington, D. C	3.52
G. W. Kavanaugh, Ithaca, N. Y......	2.53		3.70
	2.54	S. T. Donaldson, Charleston, S. C......	4.80
Miss Jessie Dent, Morgantown, W. Va.	2.61		4.76
	2.77	H. E. Curtis, Lexington, Ky	2.38
	2.80		2.43
	2.91		

These results are not satisfactory. As to the cause of the discrepancies, I have no idea and can think of nothing to recommend as an improvement.[1]

The last subject for investigation was that of the determination of the water-soluble phosphoric acid.

Here, also, we find that the causes of discrepancies may be looked for in differences in working conditions; some analysts use a filter pump and some wash to 300 cc., or even to 500 cc., the directions saying "not less than 250 cc." Now, it is well known that the so-called reverted phosphoric acid is not altogether insoluble in water, and that the longer the washing is continued, the more of this will there be dissolved. It is believed that the higher results which are obtained by washing to 300 or 500 cc. are due to the fact that some of the phosphate which is more properly reverted than soluble, has been dissolved. As it is impossible to tell exactly when all of the true water-soluble has been washed out and we are dissolving out nothing but some of the reverted, why not draw an arbitrary line? This is one of the cases where absolute accuracy is not attainable, and therefore we must be content to accept that method which will at least give the most uniformity.

Personally, I have no objection to offer, if it may seem desirable to increase the volume of washing to 300, 500 or 1,000 cc. if you please, but I believe that the volume should be definitely stated, and as there seems to be no good reason for a larger volume than 250 cc., I have asked for determinations by washing to this volume. It also seemed to me that differences were liable to ensue where one analyst used a suction pump and another did not, that in the first case the water which was rapidly sucked through did not have as great a solvent action as where it was allowed to soak into the fertilizer and drain off. In accordance with these ideas, I requested determinations of the water-soluble phosphoric acid in sample No. 1, by the following method:

Place 2 grams of sample No. 1 upon a 9 cm filter and wash with successive small portions of water, stirring it up each time by using a strong fine jet of water. Do not use a pump in this operation, but use a funnel with a good long stem and try to keep the stem full of liquid so that it will operate faster. Wash in this manner, stirring the fertilizer up with the strong fine jet of water, and allowing the solution to run off completely each time before adding the next. Collect washings in a 250 cc. graduated flask into which a few drops of nitric acid have been put, wash until within a few cubic centimeters of the mark, allow the filter to drain as far as possi-

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[1] NOTE.—I have learned from Mr. K. P. McElroy that he used an air bath at 100° C. in determining moisture. With the exception of the result of Mr. J. R. Hanahan, which is evidently too high, and those of Mr. Donaldson, which were possibly not divided by two, the remaining results are not so bad as was at first supposed, and are about as good as one could expect on moisture.

ble, remove the funnel and make up the washings to exactly 250 cc. Mix and take 62½ cc. with a pipette. Add ammonium nitrate and precipitate with molybdic mixture as usual.

The results obtained by this method are given in the first column of Table VII. The results by other methods, where pumps were used, where the washing was continued further than to 250 cc., etc., are given in column 2.

TABLE VII.—*Water-soluble phosphoric acid.*

Name of analyst.	New method.	Old method.
J. R. Hanahan, Charleston, S. C.	6.83	6.87
B. L. Murray, Geneva, N. Y	6.93	6.52
	6.88	6.55
	6.94	6.56
G. W. Cavanaugh, Ithaca, N. Y.	6.79	....
	6.82	....
	6.84	....
	6.89	....
	6.55	....
	6.63	....
	6.87	....
S. T. Donaldson, Charleston, S. C.	6.75	6.90
	6.81	6.92
F. S. Shiver, Fort Hill, S. C	6.69	....
N. Robinson, Tallahassee, Fla	6.65	....
J. A. Fries, State College, Pa	6.98	6.92
	6.93	7.01
K. P. McElroy, Washington, D. C.	6.55	....
	6.63	....
Miss Jessie Dent, Morgantown, W. Va.	6.87	....
	6.81	....
	6.74	....
	6.87	6.52
R. De Roode, Morgantown, W. Va	6.61	55 mins.
	6.65	65 mins.
	6.67	70 mins.
	6.79	85 mins.
	6.79	100 mins.
	6.81	105 mins.
	6.90	115 mins.
	6.75	120 mins.
	6.82	125 mins.
	6.88	130 mins.
	6.84	135 mins.
	6.90	145 mins.
H. E. Curtis, Lexington, Ky	6.82	6.68
	6.78	6.73
R. E. Noble, Raleigh, N. C	....	6.61
F. B. Carpenter, Raleigh, N. C	....	6.74
	....	6.77
Lehmann & Glaser, Baltimore, Md	....	6.45
A. W. Pratt, Atlanta, Ga	....	7.04
	....	6.93

It will be observed that the results in the first column show better agreement than those in the second column, although the latter are fewer in number. This is evidently due to less uniform working conditions in the latter case. The results in the first column do not, however, agree so closely as might be expected, and I feel satisfied that the differences are due to the differences in the time which was required to complete the washing. In the series of twelve determinations made by myself, which were performed side by side, I used various kinds of funnels, some operated fast, others slow, and I recorded the time necessary to complete the washing of each to 250 cc. Now, although there is some variation near the middle of the series, due to some unknown cause, the series shows quite plainly the effect of the time of washing, beginning with 6.61, 6.65 and 6.67 when the time of washing was 55 to 70 minutes and ending with 6.88, 6.84 and 6.90, when the time of washing was from 130 to 145 minutes. This element of time, then, is apparently a source of discrepancy, but how to avoid it, it is impossible to state. I think, however, that the results obtained warrant me in recommending that in the description of our methods we insert the words "do not use suction," and that instead of "not less than 250 cc.," we say "very nearly 250 cc.," or "about 250 cc."

RECOMMENDATIONS.

I feel that the amount of analytical and experimental work here reported does not justify any very material alterations of our present methods or additions thereto. There are some changes which would be advantageous, however, and I would recommend for your consideration the following:

(1) That on page 195 of Bulletin No. 35, issued by the U. S. Department of Agriculture, Division of Chemistry, the description of the method for determining water-soluble phosphoric acid be changed so as to read as follows:

"*Water-soluble phosphoric acid.*—Place two grams in a small filter, about 9 cm in diameter, wash with successive small portions of water, allowing each portion to pass through before adding more. Collect the washings in a flask graduated to 250 cc., and wash until the liquid is nearly up to the mark on the flask. Do not use suction. If the filtrate is turbid, add a little nitric acid (or a little dilute nitric acid may be added to the flask before washing is commenced). Dilute to mark, mix well, take any convenient portion, add 15 grams of dry ammonium nitrate or its equivalent and precipitate and determine the phosphoric acid as described under total phosphoric acid."

(2) That on page 195 of the bulletin referred to, the description of the method for determining citrate-insoluble phosphoric acid be so changed that it will require the citrate solution to be heated to 65° C. previous to the introduction of the fertilizer. That when it has reached 65° C. the filter containing the washed residue from the water-soluble determination be introduced. Also that the words "shake every five minutes" be inserted.

(3) That under total phosphoric acid on page 196 instead of method No. 3, the following be inserted:

"(3) Digest with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning methods for determining nitrogen. Do not add any potassium permanganate, but after the solution has become colorless, add about 100 cc. of water, boil for a few minutes, cool and make up to a convenient volume. 2.5 grams of substance and a digestion flask graduated to 250 cc. are recommended. (This method will be found convenient when both the nitrogen and the total phosphoric acid are to be determined in a sample of fertilizer.) After diluting to a definite volume and mixing, a convenient portion is sucked off with a pipette for the estimation of nitrogen. The remaining liquid in the flask is filtered through a dry filter and a portion taken for the determination of total phosphoric acid."

(4) That after the descriptions of the methods of solution for total phosphoric acid on page 196, these words be inserted:

"When the fertilizer contains much organic matter, such as cotton seed, bone, fish etc., one of the methods 1, 2, or 3 is to be used. Methods 4 and 5 are to be used only upon fertilizers containing but little organic matter, and method 6 is only to be used upon fertilizers containing much phosphate of iron or aluminium and but little organic matter."

(5) That on page 196, after the methods for total phosphoric acid, the following paragraph be inserted:

"*Available phosphoric acid.*—Subtract the percentage of insoluble from the total phosphoric acid. The remainder will be the available phosphoric acid."

Also that the next paragraph be made to read:

"*Citrate-soluble (or reverted) phosphoric acid.*—Subtract the percentage of the water-soluble from the available. The remainder will be the citrate-soluble (or reverted) phosphoric acid."

(6) That on page 196, after the description of Huston's method for preparing neutral ammonium citrate solution, in order to complete the evident idea of the author, these words be inserted: "If acid or alkaline, add ammonia or citric acid, as the case may be, mix and test again as before. Repeat the process until a neutral reaction is obtained. Now bring the solution to the proper specific gravity of 1.0900 at 20° C."

(7) That a special reporter be appointed for next year whose duty it shall be to institute investigations upon the subject of the determination of iron and alumina in phosphates.

The president called for the reading of papers which pertained to the subject of the report of Mr. De Roode.

Mr. Ross read the following paper:

DETERMINATION OF CITRATE-SOLUBLE PHOSPHORIC ACID.

By B. B. Ross.

The direct determination of citrate-soluble phosphoric acid by effecting the precipitation by means of magnesia mixture in the solution obtained from the ammonium citrate digestion, has been practiced for many years by numbers of European chemists, and the process has even obtained a place in the official methods of some European countries. Various objections have been urged, however, against the general employment of this method in fertilizer analysis on account of the inaccuracies in the results obtained in certain cases, and it has, therefore, been used to but a very limited extent in this country. Since it is impossible to effect the precipitation with ammonium molybdate in the presence of citric acid the previous elimination or destruction of this substance has been recognized as essential to the execution of a process involving the separation of the phosphoric acid as phospho-molybdate.

At the second annual meeting of this association the writer of this paper presented the results of some tests of a method proposed by him, involving the precipitation of the citric acid from the ammonium citrate filtrate by means of calcium chloride in a hot solution. This process, while yielding highly satisfactory results in many cases, gave figures somewhat too low, in other instances, on account of the precipitation of very small quantities of calcium phosphate.

The destruction of the citric acid by evaporation to dryness and ignition of the residue has also been practiced to some extent, while a writer in the Ztschr. des Ver. f. Rübenzuckerind, Nov., 1892, recommends the employment of sulphuric and nitric acid, followed by ignition, in order to effect the complete destruction of the organic matter.

A method has lately been devised in the laboratory of the Louisiana State experiment station, involving the application of the digestion method of Kjeldahl to the direct estimation of the citrate-soluble phosphoric acid. After completion of the thirty minutes digestion of the sample with citrate solution, 25 cc. are filtered out at once into a dry vessel. If the liquid is filtered directly into a dry burette, 25 cc. can be readily transferred to another vessel without dilution. After cooling, run 25 cc. of the solution into a digestion flask of 250–300 cc. capacity, add about 15 cc. of concentrated sulphuric acid and place the flask on a piece of gauze over a moderately brisk flame; in about eight minutes, the contents of the flask commence to darken and foaming begins, but will occasion no trouble, if an extremely high, or a very low flame are avoided. In about eleven to twelve minutes, the foaming ceases and the liquid in the flask appears quite black; about one gram of mercuric oxide is now added and the digestion is continued over a brisk flame. The operation can be completed in less than half an hour with ease, and in many cases, twenty-five minutes. After cooling, the contents of the flask are washed into a beaker, ammonia is added in slight excess, the solution is acidified with nitric acid and after the addition of 15 grams of ammonium nitrate, the process is conducted as usual.

In case as large an aliquot as 50 cc. of the original filtrate is used, 10 cc. of sulphuric acid are added, and the digestion is conducted in a flask of 300–500 cc. capacity; after the liquid has blackened and foaming has progressed to a considerable extent, the flask is removed from the flame, 15 cc. more of sulphuric acid are added and the flask and contents are heated at a moderate temperature for two or three minutes; the mercuric oxid is then added and the operation completed as before described.

Among the advantages offered by the method described, are:

(1) It dispenses with the necessity of the execution of the frequently tedious operation of bringing upon the filter and washing the residue from the ammonium citrate digestion; while the ignition of this residue, together with the subsequent digestion with acid and filtration are also avoided.

(2) It affords a means for the direct estimation of that form of phosphoric acid which, together with the water soluble, constitutes the available phosphoric acid; thus enabling the latter to be determined by making only two estimations.

(3) In connection with the advantages above mentioned it permits of a considerable saving of time, as well as of labor required in manipulation.

In addition to the tests with mercuric oxid, both potassium nitrate and potassium sulphate were used in the digestion to facilitate oxidation. With the former, several additions of the salt were necessary to secure a satisfactory digestion and even then the time required was longer than with the mercury or mercuric oxid digestion. With potassium sulphate, the excessive foaming which took place interfered greatly with the execution of the digestion process.

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The following are the results of practical comparative tests of estimating the citrate soluble by the proposed direct method and the usual direct method by difference:

Tests of estimating the citrate soluble.

Number.	Direct.	By difference.
	Per cent.	*Per cent.*
1...............	2.16	2.16
2...............	5.63	5.67
3...............	5.37	5.39
4...............	2.44	2.64
5...............	2.00	1.86
6...............	5.40	5.33
7...............	6.90	6.79
8...............	4.55	4.51
9...............	2.75	2.73
10...............	2.04	1.98
11...............	2.07	2.09
12...............	5.24	5.11

A number of experiments with varying proportions of pure di-sodium hydrogen phosphate, showed differences of less than .2 per cent, as compared with the direct estimation of phosphoric acids.

Mr. Ross also read the following papers:

NOTES ON THE ANALYSIS OF CONCENTRATED PHOSPHATE.

By STILLWELL and GLADDING.

During the past seven or eight years there has been such a variation in the results of the analysis of this material that we wish to present a few notes on the manner in which we have made the analysis. We have been called "high" chemists with regard to this material, and our results have been questioned by many chemists. We follow strictly the official methods as laid down from time to time.

The special point which, in our opinion, needs the most careful attention in order to obtain correct results is the neutrality of the solution. We make the citrate of ammonia solution from commercial citric acid and ammonia water. Commercial crystalized citrate of ammonia is sometimes adulterated and therefore unfit for use. Bring the citrate as near as possible to neutrality and to a specific gravity of 1.09, let it stand over night, and then complete the neutrality and determine the specific gravity.

We use as an indicator, litmus paper, which is extremely delicate and with practice we can fix exactly the neutral point. As a control, we use corallin, thus: Take 10 cc. of the citrate solution, add 10 cc. of water and a few drops of a saturated alcoholic solution of corallin. Pour the liquid from one test tube to another until it is thoroughly mixed. Divide it equally between three test tubes of equal size. Add to one tube one drop of a normal hydrochloric acid solution and to another tube one drop of a normal soda solution. If the citrate is properly neutralized there will be a marked difference between the color of the solutions in these two tubes and that of the solution in the third. One great objection to the use of corallin is that it is very difficult to obtain a sample which is sufficiently delicate for use as an indicator. Each sample of corallin should be carefully tested with standard acid and akali before using it as an indicator. We find that there is no difference in the indications of neutrality given by litmus and corallin if both indicators are of good

quality. Phenol phthalein is useless as an indicator in the presence of ammoniacal salts.

The digestion in citrate solution needs special care; first, that the "concentrated phosphate" be thoroughly moistened with the citrate solution before digestion begins; second, that the water in the bath be of such an amount that no loss of heat occurs when the flasks are added; third, that great care be taken to prevent the caking of the material on the bottom of the flasks.

For filtering, we use a plain filter of 4½ inches diameter, made of Schleicher & Schüll's paper, No. 595. Wet the paper with water and fill the stem of the funnel to secure rapid filtering. Pour the citrate solution, when taken from the bath, at once on the filter, and filter without pressure. When it has all passed through the paper, rinse out the flask four times with water at a temperature of not over 50° C., containing 10 per cent of the neutral citrate of ammonia solution. These four rinsings will fill the filter paper. Let this run through and then wash the paper once with the wash water, and when this is through put the paper with the insoluble back in the flask and dissolve and estimate as usual. The filtrate should be and will be clear if the operation is properly carried out. We use 2 grams of the material and 100 cc. of neutral citrate of ammonia, of 1.09 specific gravity.

There is nothing new in our method of working and we can not understand why so much trouble has been experienced with the analysis of this class of fertilizers. There is sometimes a variation in certain samples so that in order to obtain a sufficiently close agreement it is necessary to make from five to eight separate determinations of the insoluble. These goods, as now made, are in better condition than formerly and the variations are not so great in the analysis for insoluble. An agreement of 1 to 2 per cent in insoluble is as close as can be expected in this kind of material.

The best solvent for these iron and alumina phosphates is, for 2 grams of material, 40 to 50 cc. concentrated nitric acid plus 5 cc. of concentrated hydrochloric acid; boil 20 minuites. Use plenty of nitrate of ammonia in the precipitation by molybdate solution. The presence of the filter paper in the solution of the insoluble causes no injury. A 10 per cent solution of neutral citrate at a temperature of 45° to 50° C., used for washing the insoluble, has no appreciable solvent effect on the insoluble during the short time it is on the filter, and its use is very necessary to prevent the insoluble running through the paper.

NOTES ON THE PREPARATION OF FERTILIZER SAMPLES FOR ANALYSIS.

By STILLWELL and GLADDING.

The preparation of fertilzer samples for analysis is an operation to which too little care is given. The directions given in the official method under "Preparation of sample" simply say, "should take place as rapidly as possible so that there shall be no loss or gain of moisture during the operation."

We fear from results which have come to our notice for years past that too much is taken on trust in this part of the analysis, and that there is a gain or loss of moisture in too many cases where it is not looked for. In the analysis of blood, for example, a difference of one-tenth of 1 per cent in the ammonia contents means a difference of many dollars in the case of large lots.

It is of great importance that this matter should be officially settled by more explicit directions, and that chemists prove to themselves that there is a variation in moisture occurring during the preparation of samples. We have had many controversies with chemists who do not make the correction necessary for the variation in the moisture contents of the sample. It seldom happens that the hygroscopic condition of the air is such that no variation occurs in moisture during the fine grinding necessary to prepare the sample for analysis.

We have for over twenty years followed the practice of correcting the results of the analysis by the loss or gain of moisture incurred. We first grind or break up in a mortar the sample so that it all passes through a sieve of one-eighth or one-quarter of an inch mesh, according to the kind of material used. This can be done without any perceptible change in the moisture. The sample thus prepared we call the "original sample," and the moisture found in it—using 5 and 10 or more grams, according to the material—is the moisture of the lot under examination. A separate average of this sample is taken and finely ground for the analysis. This we call the "working sample." The moisture of this sample is also determined in duplicate. The results of the analysis of the "working sample" are calculated to a dry basis and then recalculated to the basis of the moisture found in the original sample, thereby bringing them to the true contents of the sample analyzed.

There is great need of a reform in this direction, because many disputes have arisen in the analysis of fertilizers, simply because chemists of high reputation have neglected this precaution, and manufacturers do not understand why chemists of equal skill report such varying results on precisely similar samples. On a dry basis the results may be identical, but the chemist who has corrected his results for the loss of moisture in the preparation of the sample will report lower results than are reported by one who makes no correction. It occasionally happens that in certain materials which have great absorbent power there is a gain of moisture, and the result is proportionally increased when calculated to the original moisture. If this correction be neglected, the result reported is too low, and there is complaint by the seller of the goods.

We suggest that more explicit directions be given in the official method on this point, as the matter is of great importance both to manufacturers and to chemists.

No other papers being presented, the president stated that discussion of the report of Mr. De Roode would be in order.

Mr. Huston, referring to Table II, accompanying the report by Mr. De Roode, said that there was no great accuracy in a statement of the specific gravity of the ammonium citrate solution as 1.09. Accuracy would require that it be carried out to the fourth decimal, as was done in the lower part of the same table. According to the usual interpretation, only one number in the whole series given meant anything different from 1.09. That should be corrected. In taking analytical samples for phosphate determinations, the amount weighed out was a matter of some importance. Two grams were a rather small sample. The tendency, especially among commercial chemists, was toward larger samples. He had experienced trouble in getting a certain class of goods through the sieve. Twenty per cent of the samples would not go through at all. In regard to water determination, on which he had done considerable work at high temperature, he was convinced that in superphosphates a good deal of sulphuric acid went off.

Mr. Stubbs said that he handled large quantities of English acid phosphates, which frequently came over with a guaranteed analysis, but after reaching the humid climate of Louisiana it was rare to find the moisture as low as stated. There was difficulty in weighing the acid, especially at certain seasons of year, as it gained rapidly during the process of weighing. This could be readily understood, of course, by remembering that in Louisiana it was often the case that wet-bulb and dry-bulb thermometers showed very little difference. His atten-

tion having been called to this, he had gone to some importers on the levee in New Orleans, who stated that their importations always largely overran in weight, showing that the goods in bulk absorbed a considerable amount of moisture while in transportation. They absorbed more in being sacked for shipment to plantations. He carefully ground and prepared every sample received, first milling it until it would pass through a certain sieve and then mixing thoroughly. He had found that during this process moisture was absorbed to an appreciable extent, and there was sometimes great difficulty in weighing with sufficient rapidity to insure accuracy. He thought it would never be possible for samples sent all over the United States to agree in respect to the determined moisture.

Mr. CHAZAL believed that the methods for moisture determination given in the reports of the association were susceptible of great improvement. Those who had to deal almost entirely with fertilizers, and handled largely ammoniated material, did not have to fully face the difficulty he encountered. He had found that there was frequently a loss of 3, 4, or 5 per cent of moisture in grinding a sample of tankage so that it would pass through the prescribed sieve. His analyses were made on the ground material, and if that analysis were sent in the fertilizer consumer would have to pay for more nitrogen than he got. It was therefore necessary to grind as rapidly as possible, draw a sample of the material, determine the moisture on a large quantity, 5 to 10 grams, determine the moisture on a finely ground sample, and reduce it from one sample to the other. The rules of the association made no provision for this, and it was important. When he had only a high-grade black blood no moisture was lost, but now that everything was being used the matter became one of great importance. It would confer a great favor on commercial chemists if the association would adopt rules covering the case. To do away with the use of the pump in determination of the water-soluble acid, as recommended by the reporter, would be a hardship to commercial chemists, who find the pump of great assistance; unquestionably, it greatly hastens the work. He sometimes had to handle acid phosphates almost green, some of which required three or four hours to run through the filter without the aid of a pump. Unless some strong reason existed to show that better results were obtainable without the pump, he hoped it might be retained. The results obtained by Mr. De Roode, using the old method, were not sufficiently numerous—he would not say accurate—to prove anything.

Mr. VAN SLYKE moved that the recommendations of the reporter be taken up and considered in their order.

The motion was carried.

Mr. DE ROODE read again his first recommendation, and added: I have said here, "Do not use suction." I will withdraw that, or put it to the vote of the association. There is now a tendency to use pipettes which will deliver a definite volume, thus saving calculation. As I

will change the recommendation, the first question raised is whether we shall wash to a definite volume of 25) cc., or not less than 250 cc., leaving it to the discretion of the analyst whether he shall add more than that. I move that the method to be adopted shall direct as follows: Collect the washings in a flask graduated to 250 cc., and wash until the liquid is exactly up to that mark.

The motion was seconded.

Mr. FREAR suggested that the direction be to wash until the wash. ings amount to nearly 250 cc.

Mr. CHAZAL had supposed that the method provided for exactly 250 cc. It had formerly been "not less than 250 cc.," but he thought it had been changed so as to call for just 250 cc. Mr. De Roode had called attention to a point which he seemed to have overlooked in his recommendation. It was frequently necessary to add nitric acid, and some chemists would go up to 250 cc., while some stopped short of that. For 250 cc. any flask could be used.

Mr. DE ROODE said that he desired the amount specified to be 250 cc. or very nearly that.

Mr. CHAZAL thought that "nearly" was a very uncertain term.

Mr. DE ROODE stated that the uncertainty was what he intended to avoid. In order to make the matter clear, he was willing to adopt any change made by vote of the association. He renewed his motion to substitute exactly 250 cc. for "nearly 250 cc." in the method of determining water soluble phosphoric acid (Bull. 35, p. 195).

Mr. STUBBS said that a great deal depended upon the rapidity of the washings and upon the size of the funnel used. The size of the funnel should be defined.

Mr. McDONNELL thought it would be well to prescribe a 9 cm. filter.

Mr. DE ROODE. I will adopt Mr. McDonnell's suggestion and change my recommendation so that instead of reading "Place 2 grams in a small filter, about 9 cm. in diameter," it shall read as follows: Place 2 grams in a 9 cm. filter. I move that this change be made.

The motion was carried.

Mr. DE ROODE. The method as now proposed will be: Wash with successive small portions of water, allowing each portion to pass through before adding more. Collect the washings in a flask graduated to exactly 250 cc.

Mr. McDONNELL. I think the word "exactly" is rather superfluous there.

Mr. DE ROODE. Then let it say until the filtrate measures 250 cubic centimeters.

Mr. FREAR said that the exact quantity could not be measured. In a careful experiment he had made with some moderately green goods he had added somewhat more than 100 cubic centimeters after washing to 250 with a practically identical result. Whether or not the quantity overran made some difference in the convenience of handling. Probably

any chemist would understand the direction as meaning that he should come as near to the quantity as he could. He believed, however, that the direction given was sufficiently definite for practical work.

Mr. LORD believed that his own experience in regard to washing would be confirmed by the experience of others. The purification of the precipitate depended more upon the number of times it was covered with water and allowed to drain than upon the absolute amount of water run through it.

Mr. HUSTON objected to the words "exactly 250 cubic centimeters." He knew that his assistants would not make the filtrate up to that amount unless he watched them, which he did not care to do. He agreed with Mr. Frear that the last few (say 5) cubic centimeters cut but a very small figure. Five cubic centimeters was about as small a quantity as would be added at any one washing, and the addition of the last 5 cubic centimeters in the long series of washings amounted to nothing when compared with the sample originally weighed. He thought it would be well within the margin of safety to direct that the filtrate should finally measure from 245 to 250 cubic centimeters, which would leave some room for the nitric acid and a safety valve for the termination of the washing.

Mr. DE ROODE said that he had carefully considered the point suggested before making his recommendations. In answer to an inquiry by Mr. Chazal, he said he would be willing to have the direction read between 240 and 250 cubic centimeters.

Mr. CHAZAL made the point that many analysts had a great deal of apparatus on hand, which it would be expensive and inconvenient to change. A difference of 10 or even 20 cubic centimeters would not make a particle of difference in the results. The weighing was really the matter of most difficulty.

Mr. DE ROODE. I will amend my recommendation and the motion thereon so that the direction shall be to wash until the liquid measures about 250 cubic centimeters. Then if anyone wishes to wash a little less he will be at liberty to do so.

The motion to adopt the recommendation, as amended, was carried.

Mr. DE ROODE. I move the adoption of my next recommendation, which is, "Do not use suction."

Mr. CHAZAL, as a commercial chemist, wished to call attention to the difference between the work of commercial and official chemists in the matter of suction. The latter could begin the analyses of twenty or thirty samples at once, and it made no difference whether the suction pump were used or not; but to a commercial chemist working upon one sample, and the analysis having to be made in a hurry, the difference was greatly in favor of the suction pump. The difference was not a question of principle, but only of uniformity, and if official chemists had to commence their many analyses separately, and wait while the

water soluble ran through, they would hesitate before depriving commercial chemists of the use of the suction pump.

Mr. HUSTON said that some official chemists had to work under the same conditions that confronted the commercial chemists, especially in the strict inspection States. Orders were received by telegraph, and in filling them it was often necessary to work as rapidly as did the commercial chemists, so that it was frequently important to get the analyses started promptly. Until definite numerical information could be had upon the question whether the use or nonuse of the filter pump made a difference, chemists should be allowed to use their own discretion in this regard. He would be very unwilling to abandon the use of the filter pump in determining water soluble, and believed that there was really a matter of principle involved—that the washing could not be done as well without it as with it.

Mr. DE ROODE replied that he had made his recommendation for the very reason that commercial chemists and official chemists who, like Mr. Huston, worked in a hurry, got results which did not agree with those of other chemists. It was more important to make the determination accurately than to make it quickly.

Mr. CHAZAL thought the last statement hardly fair. While he could not answer for other laboratories, he was sure that in his own no method had been employed for the mere sake of haste; but as between two methods giving equally good results, he did prefer the quicker one. If it could be shown that inaccuracy resulted from the use of the suction pump, he would be willing to devote hours to the operation. He felt, however, that until the association had more definite figures to prove the point made against the pump the method should remain unchanged.

Mr. SCOVELL said there were no figures to show that the method should be changed as recommended, while there were figures showing that the filter pump did the work more accurately than it was done by the manner proposed. The association could not go back without good reason. The pump had been adopted on account of the uniformity it insured. At least it had been recommended, but some chemists had none, and so the method had been allowed to stand as it was, leaving the use of the pump optional. The results shown in former reports, however, seemed to prove that determinations made with the use of the pump were quite as accurate as those obtained by hand, if not more so.

Mr. DE ROODE. I have here some figures by chemists in Mr. Scovell's own laboratory, which show that they obtained higher results without the filter pump. All the figures I have given show that the results are higher without the pump than with it.

Mr. HUSTON said that the explanation of that fact was a very reasonable one. Reverted phosphoric acid was soluble in a solution of water-soluble phosphoric acid. He believed that the very matter of allowing the slow filtration to proceed was the explanation of the figures given.

The motion to adopt the recommendation in question was lost.

Mr. DE ROODE. My next recommendation is as follows:

If the filtrate is turbid, add a little nitric acid (or a little dilute nitric acid may be added to the flask before washing is commenced).

The motion is that those words be inserted. We all do that, but it has not been prescribed.

The motion was adopted.

Mr. DE ROODE. I next say—

Dilute to mark, mix well, take any convenient portion, add 15 grams of dry ammonium nitrate or its equivalent and precipitate, and determine the phosphoric acid as described under total phosphoric acid.

Instead of "Dilute to mark" I will make it read, "Dilute to some definite volume." I have said "any convenient portion" instead of "an aliquot portion," which calls for an exact multiple.

Mr. HUSTON. I think it would be a good idea to fix a minimum to that "convenient portion."

The PRESIDENT. In the present method the direction is to take an aliquot portion corresponding to one-half a gram.

Mr. McDONNELL. It should be, I think, "take an aliquot part." It certainly is so stated under total phosphoric acid, if not under the soluble.

Mr. PARSONS. If there is no objection, I think it very much preferable to leave it as it is, for the reason that there are hardly any of us who do not use the tables of Dancy and Battle for calculations.

Mr. McDONNELL. I use 0.4 of a gram, and prefer to use a table constructed by myself.

Mr. DE ROODE. The words "any convenient portion" would cover that case.

Mr. ROSS. You might also say "an aliquot part of the whole."

Mr. DE ROODE. Then you would have to use some multiple or submultiple.

Mr. FREAR. We ought not, I think, to seem to favor taking too small a quantity of material. That is a point as to which the judgment of some chemists can not be altogether trusted, especially when they are to compare their work with ours. The limits of error in a very small quantity are much wider than in a large quantity.

Mr. DE ROODE. I have found a superphosphate, manufactured by a company in Baltimore, that contains 45 per cent of available phosphoric acid. The portion corresponding to 0.5 gram is too large in that case. If we say "any convenient portion," any chemist can use his judgment about that.

The motion was put. The vote was a tie.

Mr. CHAZAL. Suppose it be "not less than 0.5 gram in the ordinary standard goods?"

Mr. DE ROODE. That is satisfactory.

Mr. FREAR. Perfectly so. It seems to me that in analyzing the same grade of goods we should use methods capable of commercially exact comparison.

Mr. HUSTON. I would call attention to the fact that if we let it alone there can be no harm done. If, however, a change is to be made, I think we had better adopt the wording found on page 196 of the bulletin, under "Total phosphoric acid:"

* * * take an aliquot part of the filtrate corresponding to 0.25 gram, 0.50 gram, or 1 gram.

I am not sure that is not the best thing to do with this. We can then work on low or very high solubles.

Mr. DE ROODE. But that does not allow the use of these very convenient pipettes which have been introduced by the New Jersey station. They are very good things, and I think their use ought to be allowed.

Mr. SCOVELL. I am in favor of leaving as much as possible to the judgment of the chemist. Any ordinary chemist knows how much to use, or, if he does not get it right the first time, he will try it over again. I know that they will do it that way in New Jersey whether we adopt it or not.

The motion was again put, and was carried.

Mr. DE ROODE. The next recommendation is that we add 15 grams of dry ammonium nitrate or its equivalent.

Mr. CHAZAL said that if a man did not wish to follow the official methods he would not do so, whatever recommendations were made and adopted. New Jersey chemists made their analyses according to New Jersey law, and had never paid any attention to the methods of the association. If outsiders wished to add ammonium nitrate they would do so. The only object of the association was to prescribe methods that all would follow, but no method could be adopted which a chemist would not modify if he chose. He did not see the slightest advantage in the addition of the ammonium nitrate, but deemed it a waste of time and material, and hoped the change would not be made.

Mr. DE ROODE thought that the opinions of those who did add the ammonium nitrate should not be ignored. He would be glad to have its advantages stated by those who used it.

Mr. GAINES said that it brought down the phosphoric acid precipitate in a very few minutes. One could go ahead with his determination of phosphoric acid in fifteen minutes and get accurate results. In his own experience, without the ammonium nitrate, it had taken two hours for the phosphoric acid to come down.

Mr. CHAZAL said that the official method did not allow filtration after fifteen minutes; it required digestion for at least one hour, and it was only rarely that the soluble did not come down in that time.

Mr. DE ROODE had found that sometimes, without the addition of the ammonium nitrate, the soluble did not come down in so good a condition. He did not urge that fact as one showing an especial advan-

tage in the use of the ammonium nitrate, but would like to have others who used it present stronger arguments in its favor.

Mr. FREAR had tried it two years ago, and it made no appreciable difference. He put it in merely for the fun of the thing.

Mr. CURTIS said that it was used in Kentucky to more easily precipitate the filtrate; if it were not put in, the filtration required twice the time.

Mr. CHAZAL saw no objection to the use of the nitrate, but there was no advantage in being compelled to use it. He thought it made no real difference, but that it would be unwise to insert it in the method.

Mr. DE ROODE. I will change my recommendation so that it shall read, "Add 15 grams of dry ammonium nitrate or its equivalent if deemed necessary." Then it can be used or not.

On motion of Mr. Huston, the matter of the addition of the ammonium nitrate was referred to the reporter on phosphoric acid for the next year.

Mr. DE ROODE read his next recommendation, as follows:

That on page 195 the description of the method for determining citrate-insoluble phosphoric acid be so changed that it will require the citrate solution to be heated to 65° C. previous to the introduction of the fertilizer. That when it has reached 65° C. the filter containing the washed residue from the water-soluble determination be introduced.

Mr. HUSTON was opposed to changing the present method unless a better one could be substituted for it. The method for determining insoluble in goods the base of which consisted entirely or almost entirely of calcium was as good as any other, but was little or no better than the old one in case of goods, now coming largely upon the market, which contained higher amounts of alumina, and were mixtures of finely ground alumina and iron with tankage, the alumina never having been acidulated. After working for a number of years upon that class of goods, he was convinced that they contained no reverted phosphoric acid. Nearly all of the phosphoric acid in these goods had practically the same agricultural value. As he understood, there was no complaint in regard to pure calcium goods, the trouble being always due to relatively large amounts of iron and alumina, and if the association undertook to reach that case it would have to adopt more radical measures than were now proposed. All of the phosphoric acid in phosphate of iron and alumina would dissolve in ammonium citrate under any of several conditions. The soluble could be increased by increasing the temperature or rendering the solution alkaline. The question of reverted determinations in all such goods was still to be solved, and an additional digestion of five to fifteen minutes had very little to do with the case. It was physically impossible to interrupt a digestion when a solution was going on at any such rate and get accurate results. The present method had been in use for a long time and ought not to be changed. On goods easily worked there would be no practical difference by the use of the proposed method, and it would afford no improvement in the case of goods which were worked with difficulty

Mr. De Roode did not claim that the change recommended would effect any improvement in the determination of citrate-soluble phosphoric acid in the class of goods referred to by Mr. Huston. His only claim of advantage over the old method was that it introduced a little more uniformity into the working directions.

Mr. Frear said that some of the results reported by Mr. Williams, of the Franklin Institute, about eight or nine months ago, at the time when Mr. Terne discussed the work of the association, seemed to indicate that differences in size of bath, etc., led to difference in time, and in certain classes of goods to difference in results.

Mr. De Roode. My recommendation is based on the results obtained in work done this year. These are much more uniform than those obtained by the old method.

The recommendation was not adopted. (During the proceedings of the second day Mr. Kedzie moved to reconsider this vote, and after further discussion the recommendation of the reporter was adopted.)

Mr. De Roode. I move that the words "shake every five minutes" be inserted.

Mr. Gaines. That would be very indefinite.

Mr. De Roode. I mean that those words should be inserted on page 195, following the direction—

After the introduction of the flasks the temperature of the bath should be brought as rapidly as possible to 65° and maintained at this point for thirty minutes.

The motion was carried.

Mr. De Roode. My next recommendation is as follows:

Digest with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning methods for determining nitrogen. Do not add any potassium permanganate, but after the solution has become colorless add about 100 cc. of water, boil for a few minutes, cool, and make up to a convenient volume. 2.5 grams of substance and a digestion flask graduated to 250 cc. are recommended. (This method will be found convenient when both the nitrogen and the total phosphoric acid are to be determined in a sample of fertilizer. After diluting to a definite volume and mixing, a convenient portion is sucked off with a pipette for the estimation of nitrogen. The remaining liquid in the flask is filtered through a dry filter and a portion taken for the determination of total phosphoric acid.)

That is intended to go in as the third direction under total phosphoric acid, page 196.

Mr. Kilgore said that all sulphuric acid methods should be prescribed with limitations. On goods high in calcium phosphate the precipitate was liable to bring down some sulphuric acid, and some workers had complained of that. He had experienced some trouble of this kind, though he had not followed the method very closely, and the same thing had been mentioned in the journals. With ordinary fertilizers, containing only 8 to 12 per cent of phosphoric acid, there would probably be no difficulty, but there was danger in the case of goods high in calcium phosphate.

The reporter's recommendation was adopted.

Mr. De Roode. I would further recommend that after the descriptions of the methods of solution for total phosphoric acid, on page 196, the following be inserted:

When the fertilizer contains much organic matter, such as cotton seed, bone, fish, etc., one of the methods 1, 2, or 3 is to be used. Methods 4 and 5 are only to be used upon fertilizers containing but little organic matter, and method 6 is only to be used upon fertilizers containing much phosphate of iron or aluminum and but little organic matter.

Mr. Huston. I would say, after method 6, "This method is especially recommended for goods high in alumina and iron."

Mr. De Roode. Would you say anything about methods 4 and 5?

Mr. Huston. I do not think it is necessary, for the reason that there is already present, at the end of method 3, the statement that—

These methods are to be used in case of fertilizers containing cotton seed or much organic matter.

Mr. De Roode. I will withdraw the recommendation.

Mr. Hartwell. Going back to the citrate insoluble, the first direction is as follows:

Wash the residue in the filter from the above determination into a 200 cc. flask with 100 cc. of strictly neutral ammonium-citrate solution.

I would like to know just how that is to be done, as a matter of information.

Mr. De Roode. I do not know. I can not wash the residue off a porous filter. Some of it sticks in the filter. I always put the filter in first. I do not believe that anybody can do it, though some claim that they can.

Mr. McDonnell. It is easily done if you spread the filter on a glass plate. In some cases it is necessary to rub a part of the filter off with the finger; but only in a very few cases have I found it necessary to introduce the whole filter.

Mr. De Roode. We have no description of a method for calculating the available phosphoric acid. I recommend that on page 196, after the methods for total phosphoric acid, the following paragraph be inserted:

Available phosphoric acid.—Subtract the percentage of insoluble from the total phosphoric acid. The remainder will be the available phosphoric acid.

I also recommend that the next paragraph be made to read as follows:

Citrate-soluble (or reverted) phosphoric acid.—Subtract the percentage of the water soluble from the available. The remainder will be the citrate-soluble (or reverted) phosphoric acid.

Mr. Stubbs. We discussed that subject years ago and dropped all that had been said about available phosphoric acid. In our State the law does not recognize it. We leave it for the farmer to judge as to what is available.

The recommendations were not adopted.

Mr. De Roode. I recommend that on page 196, after the description of Huston's method for preparing neutral ammonium-citrate solution,

in order to complete the evident idea of the author, the following words be inserted:

If acid or alkaline, add ammonia or citric acid, as the case may be; mix and test again as before. Repeat the process until a neutral reaction is obtained. Now bring the solution to the proper specific gravity of 1.0900 at 20° C.

The recommendation was adopted.

Mr. DE ROODE. My last recommendation is as follows:

That a special reporter be appointed for next year, whose duty it shall be to institute investigations upon the subject of the determination of iron and alumina in phosphates.

There seems to be some disagreement among the members of this association as to whether that is within the province of the work we are doing. I would like to have that question decided. If it be declared that this matter is within the scope of our labors, some measures should be taken which will render it unnecessary for the reporter on phosphoric acid to do all that work.

Mr. CHAZAL. That is a very serious question. It is only rarely that the official chemists have much to do with oxid of iron and alumina. The commercial chemists are forced to use the test a great deal, and must employ such methods as will enable the sale of phosphate rock in Europe; it is no longer sold in this country. I mean to say that all the methods are regulated by the European market and the European chemists. We wish to use official methods, and have to print upon our blanks a statement that we do so; but we can not undertake to use official methods on oxid of iron and alumina. I think it is unwise for the association to go beyond its tether.

Mr. McDONNELL. I hardly think that this association is ready at the present time to take up that question. As Mr. Chazal has said, it does not seriously concern the members of our association, but does greatly concern the trade chemists.

The recommendation was not adopted.

Mr. HUSTON. I would like to call attention to the last three lines in the method of determining citrate-insoluble phosphoric acid, on page 195. The direction there given is as follows:

In case the substance contains much animal matter (bone, fish, etc.), the residue insoluble in ammonium citrate is to be treated by one of the processes described below under "Total phosphoric acid," 1, 2, or 3.

If we make that read 1, 2, 3, or 4, it will be complete.

Mr. DE ROODE. That should be done.

Mr. KILGORE moved that the change suggested be adopted.

Mr. SCOVELL. If I recollect right, that same method came up before us two years ago, and we had no data upon which to change the method. I think the question was broached by Mr. Voorhees, who had tried it, treating the filter with hydrochloric acid. He did not succeed, and there was some evidence that it would not give accurate results with the filter paper. I would like to see figures before that change is made. I think it would be well to refer it to the reporter.

Mr. Kilgore's motion was withdrawn.

On motion of Mr. Frear, the matter under discussion was referred to the reporter for next year.

Mr. Ross moved that the reporter on phosphoric acid, for the ensuing year, be directed to test the applicability of the Kjeldahl method to the direct determination of citrate-soluble phosphoric acid.

The motion was carried.

Mr. PARSONS. I think that all the suggestions we have to make should be printed in our proceedings, but that the reporters should be allowed to use their own judgment to a considerable extent when it comes to the sending out of samples.

On motion of Mr. Kedzie, the recommendations of the reporter on phosphoric acid, as amended, were adopted.

The discussion of the phosphoric acid report having been finished, Mr. Parsons was called on to read the report on nitrogen.

REPORT ON METHODS FOR THE DETERMINATION OF NITROGEN.

By CHARLES LATHROP PARSONS.

Thanks to the motion made by one of our number, the reporter on nitrogen has this year been relieved of a very laborious part of his usual duty, that of abstracting the nitrogen literature of the year for presentation in his report, and this work having been referred to a special committee, I have not entered upon any attempt at a bibliography on this subject.

By vote of the association at its last meeting the nitrogen reporter was this year instructed to study into a method whereby the Gunning method could be adopted for use with fertilizers containing nitrates; that the potassium tetraoxalate method for standardizing alkaline solutions be given further trial, and that a method for the standardization of such alkaline solutions, based on a comparison of such solution with a quarter-normal solution of sulphuric acid determined by precipitation as $BaSO_4$, be investigated. In correspondence with many chemists interested in the work, I found a lingering doubt in the mind of a few of the complete accuracy of the Gunning method as compared with that of Kjeldahl, and I decided to give these two methods a further comparison.

In pursuance of these instructions and ideas I prepared two samples for nitrogen determination, as follows: No. 1, ground cow peas; No. 2, a home-made fertilizer containing the following constituents: cotton-seed meal, dried blood, potassium nitrate, dissolved bone black, and muriate of potash. The potassium nitrate made up just one-fourth of the weight of the mixture, and the total nitrogen was calculated to be about 7 per cent. Analysis has shown that some of the constituents were unusually poor in their nitrogen contents, and that the total actual amount present was somewhat less than 6 per cent.

Both samples were ground according to the official method, and while this grinding was sufficient in the case of the cow peas it may be that sample No. 2 would have been better if finer ground. The samples were most carefully mixed by placing each in a box mounted on trunnions, so that as the box was slowly turned the sample would continually fall from one corner to the corner diagonally opposite, being in this manner continually thrown in three different directions. This turning was kept up for several hours, and it is my opinion that the mixture at the time of bottling was as perfect as it could well be made. All the bottles were filled at the same time, carefully corked, and sealed with paraffin. They were only about two-thirds filled in order that they might be again mixed by turning if any silting

of the finer particles to the bottom took place during transportation. Besides these two samples a 300 cc. bottle of standard HCl and an ounce bottle of Merck's guaranteed potassium tetraoxalate, imported specially for this purpose, were sent to each chemist. A letter of instructions embracing the following points was sent out at the same time:

"(1) Sample No. 1, consisting of ground cow peas, you are requested to analyze by both Kjeldahl and Gunning methods (pp. 200 and 202 of Bulletin 35, U. S. Department of Agriculture, Division of Chemistry) as a further comparison of these methods in the hands of different analysts.

"(2) Sample No. 2, consisting of a fertilizer containing about 7 per cent of nitrogen, of which about one-half is in nitrates and the remainder easily decomposable organic nitrogen, you are requested to analyze by the Scovell-Kjeldahl method (p. 202, Bulletin 35) and by the Gunning method modified for nitrates as proposed by Mr. E. B. Voorhees (p. 86, Bulletin 35), and besides to determine if sodium thiosulphate, which would introduce no heavy metals into the solution, may not advantageously be substituted for the zinc sulphid recommended.

"(3) It seems very desirable, in view of the greater number of analyses that can be carried on by the Ulsch method at one time than by the Schulze-Tiemann method, and its general simplicity and easy adaptability to our ordinary Kjeldahl apparatus, that some modification of it meeting our needs for the accurate estimation of nitrogen in nitrates should be found. You are therefore further requested to analyze sample No. 2 for nitrates by the method proposed by Mr. Street on page 89 of Bulletin 35. The original Ulsch method, of which this is a modification, may be found on pages 175–182 of the Ztschr. anal. Chem., **30**, 1891.

"Please report as follows on the standard acid and as much in addition as your time and inclination will permit:

"(a) The amount of HCl in 100 cc. of the standard acid, determined by precipitation as AgCl, as directed on page 200 of Bulletin 35.

"(b) The number of cubic centimeters of your ammonia solution equivalent to 20 cc. of the standard acid.

"(c) The strength of your ammonia solution determined by means of potassium tetraoxalate (mol. wt. 253. 52).

"(d) The strength of the standard acid based on a calculation from data obtained in b and c.

"(e) The strength of your ammonia solution by the sulphuric acid method. (See below.)

"(f) The strength of the standard acid based on a calculation from data obtained in b and e.

"In determining the strength of your ammonia solution by potassium tetraoxalate, approximately 0.2 grams should be carefully weighed on a watch glass, transferred to a beaker, and dissolved by adding to it 100 cc. of recently boiled hot distilled water. On cooling add litmus solution (prepared as directed in Sutton's Volumetric Analysis, page 30, 6th ed.) to suit the eye of the analyst, and titrate with the ammonia solution until a *blue* color appears. In this connection please read the paragraph beginning with the word "Litmus," on page 84 of Bul. 35, and if the tetraoxalate does not prove to be pure $KHC_2O_4H_2C_2O_4+2H_2O$, recrystallize as directed on page 83.

"For determining the strength of the ammonia solution by comparison with standard sulphuric acid, the gentleman who moved that this method be investigated describes his mode of procedure to me substantially as follows: Add 15 cc. C. P. H_2SO_4 to 2 liters of distilled water. Determine strength of this solution by precipitating 10 cc. of it, measured from an accurately calibrated pipette, by $BaCl_2$. Make four or five determinations which should agree closely, and take the mean. Keep this acid solution in a tightly stoppered bottle in a cool dark place and determine strength of other acid and alkali solutions by titrating against this as a standard.

"Please see that in all this work on standard solutions that when the titrations are

made the solutions are at or near the temperature of a comfortable working laboratory, *i. e.*, about 20°.

"It is, perhaps, unnecessary in closing to add the caution that all chemicals be carefully tested for purity by blank experiments; that the bottles be kept sealed until the work is begun; and that the samples be carefully remixed by a prolonged turning of the bottles before unsealing as a precaution against the sifting out of the finer particles. Especially on sample No. 2, I would recommend that the bottle be turned a few times each time before weighing out samples."

Seventy-two chemists were requested to coöperate, twenty-nine agreed to do so, and twenty-one sent in complete or partial reports.

RESULTS OF ANALYSIS.

Moisture.—The few results reported on per cent of water in both samples No. 1 and No. 2 were so nearly identical as to leave little doubt that they had been securely sealed and that their water content was unchanged. The averages reported were: Moisture in No. 1, 14.75; moisture in No. 2, 2.15.

Sample No. 1—cow peas.

From—	Analyst.	Per cent of nitrogen.	
		Official Kjeldahl method.	Official Gunning method.
U. S. Dept. of Agriculture, Chemical Division..	T. C. Trescot	3.20	3.24
U. S. Dept. of Agriculture, Biochemic Laboratory.	J. A. Emery..........	3.32	3.31
Maryland Exp. Station.........................	H. J. Patterson.......	3.23	3.26
Maryland College.............................	H. B. McDonnell......	3.25	3.20
Wisconsin Exp. Station	F. W. Woll...........	3.11	3.09
New Jersey State Exp. Station.................	E. B. Voorhees	3.29	3.27
Mass. State Exp. Station......................	C. H. Jones..........	3.29	3.24
New York Exp. Station, Geneva...............	W. B. Cady..........	3.29	3.23
	A. L. Knisely	3.30	3.28
Maine Exp. Station...........................	L. H. Merrill	3.34	3.25
Nebraska Exp. Station	T. L. Lyon	3.22	3.23
North Carolina Exp. Station..................	F. B. Carpenter	3.25	3.27
Charleston, S. C	S. T. Donaldson	3.27	3.26
	J. R. Hanahan........	3.34	3.32
New Hampshire Exp. Station	F. W. Morse..........	3.36	3.27
	E. P. Stone...........	3.32	3.28
New Hampshire College.......................	C. L. Parsons	3.30	3.26
Average		3.28	3.25

The absolute method gave Mr. Trescot 3.30, and the Ruffle method gave Mr. Carpenter 3.20.

DISCUSSION OF RESULTS ON COW PEAS.

The results, with one exception, are very close. All chemists reported themselves as much pleased with the Gunning method or did not mention the matter at all. No adverse criticisms were received, and your reporter has no comments to record. Omitting one result in making the summary it stands as follows:

Number of analysts	16
Highest result ....per cent..	3.36
Lowest result......................................do...	3.20
Average of all results.................................do...	3.28
Greatest variation from average.......................do...	.08

For sixteen different chemists working at different times, at different places under varying conditions of temperature and with acid and alkaline solutions varying, as will be shown, widely in strength, little better can be asked. It would seem, therefore, that these results should settle definitely the question as to whether the Gunning method can be safely used on our ordinary fertilizers and food stuffs as a means for determining their nitrogen contents instead of the Kjeldahl method, and I believe that the cleanliness, easy distillation, and smaller number of reagents required by this method will lead to its general adoption by American agricultural chemists.

Sample No. 2—fertilizer.

From—	Analyst.	Per cent nitrogen.	
		Official Kjeldahl modified for nitrates.	Gunning modified for nitrates.
U. S. Dept. of Agriculture, Division of Chemistry.	T. C. Trescot	5.96 [1]5.92	5.90 [1]5.97
U. S. Dept. of Agriculture, Biochemic Laboratory	J. A. Emery	[3][5]5.93 [1]5.93	5.90 [1]5.92
Maryland Exp. Station.........................	H. J. Patterson	[4]6.01 [1]6.00	[7]6.38 ....
Maryland College.............................	H. B. McDonnell......	5.83	5.79 [1]5.61
Wisconsin Exp. Station.......................	F. W. Woll	[3]5.59	[5]5.69 [1]5.63
New Jersey Exp. Station......................	E. B. Voorhees........	5.89	5.86 [1]5.85
Mass. State Exp. Station......................	C. H. Jones...........	5.88 [1]5.80	([9]) ....
Connecticut Exp. Station ....:...............	A. L. Winton	5.96 [1]5.91	[2]5.98 [1]5.95
Geneva, N. Y., Exp. Station...................	W. B. Cady	5.74 [1]5.68	5.72 [1]5.55
Do...	A. L. Knisely	5.74 [1]5.72	5.62 [1]5.62
Maine Exp. Station...........................	L. H. Merrill..........	5.95 [1]5.94	....
Nebraska Exp. Station	T. L. Lyon	5.69 [1]5.58	5.65 ....
North Carolina Exp. Station..................	F. B, Carpenter	5.78	....
Charleston, S. C.............................	S. T. Donaldson.......	[4][7]6.33	[4][7]6.23
Do...	J. R. Hanahan........	6.09	....
New Hampshire Exp. Station	F. W. Morse	[1]5.68	[1]5.68
Do...	E. P. Stone............	[1]5.70	[1]5.74
New Hampshire College.......................	C. L. Parsons	5.78	[6][7]6.30 [1]5.85
Average......................................		5.77	5.77

[1] Use of $Na_2S_2O_3$.

[2] Digestion at low temperature, then addition of K_2SO_4 and high temperature.

[3] No ZnS or $Na_2S_2O_3$ used.

[4] ZnS used in both cases, which explains discrepancy.

[5] Sample reground.

[6] No correction for blank amounting to 1.5 cc. HCl. Corrected result, **5.80.**

[7] Not included in average.

[8] Corrected result. ZnS gave blank of several cc. acid solution.

[9] Unsatisfactory results on account of impurity of ZnS.

The absolute method gave Mr. Trescot 5.90.

Omitting all results marked ([7]), the official Kjeldahl modified gives as highest 6.09 per cent, as lowest 5.58 per cent.

The greatest variation from average being 0.32 per cent.

The variation from average is not so great where $Na_2S_2O_3$ is used, but too great to be satisfactory. The results by the same chemist using zinc dust or sodium thiosulphate agree well.

Omitting all results marked ([7]), high in every case on account of impurities in ZnS, the Gunning method modified gives as highest 5.98 per cent, as lowest 5.55 per cent.

Where ZnS is used, difference between highest and lowest 0.76 per cent.

Where $Na_2S_2O_3$ was used, difference between highest and lowest, 0.42 per cent.

COMMENTS OF DIFFERENT ANALYSTS.

F. W. Woll.—Zinc dust was used in case of Kjeldahl-Scovell method. First sample of zinc sulphid tried required a correction of 7.94 cc. HCl. Origin unknown. Some of Merck's zinc sulphid required for three grams a correction of 2.32 cc. HCl. It would therefore seem that caution is in order in regard to its use.

E. B. Voorhees.—The improved Gunning method for the determination of total nitrogen in samples containing nitrates and the Ulsch method for the determination of nitrates have proved entirely satisfactory in the regular work of the laboratory.

J. B. Lindsay.—With modified Gunning method we were not successful. Found difficulty in procuring zinc sulphid free from nitrogen compounds. Two different samples both contained nitrogen, both as nitrates and ammonia salts. In sample furnished by you neither nitrates nor ammonia salts could be found. Still our results were invariably higher than by either other method, nearly 1 per cent. Ulsch method modified by Street yielded from 2.10 to 2.36 per cent nitrogen.

H. B. McDonnell.—Modified Gunning with sodium thiosulphate foamed badly.

A. L. Winton.—In each case the material was thoroughly shaken and allowed to stand an hour or two with repeated shakings. The reducing agent was then added and the mixture heated over a low flame for a few minutes. After adding the oxidizing agent the digestion was finished at a higher temperature. Working in this manner the following results were obtained:

10 grams potassium sulphate.	Sample No. 2.	Potassium nitrate.
	Per cent.	*Per cent.*
2 grams zinc dust.............	5.96	13.69
3 grams zinc sulphid	5.98	13.72
5 grams sodium thiosulphate...	5.95	13.77

Much of the zinc sulphid prepared by manufacturing chemists contains a considerable amount of ammonia, and it is therefore safer to use only the mineral zinc blende.

F. B. Carpenter.—Attention is called to the use of zinc dust instead of potassium sulphid in both the plain and modified Kjeldahl methods.

	Plain Kjeldahl.	Modified Kjeldahl.
	Per cent.	*Per cent.*
K_2S............................	3.25	5.78
Zinc dust, 2 grams..............	3.24	5.77

In studying the results for total nitrogen on sample No. 2 the first fact which strikes the attention is the discrepancies which exist, while in general there is a very close

agreement between the different results obtained by the same chemist. The very adverse results obtained by the use of zinc sulphid are also strikingly apparent. The results are by no means what they should be for anything like accurate work, and their variance is due, in my opinion, to three reasons, with the possible influence of a fourth. These reasons are as follows, arranged in the inverse order of their importance:

(1) A tendency on the part of the chemists, unacknowledged even to themselves, to see the neutrality point in titration a drop or two too soon or too late, if it will make results agree more closely. This can easily be overcome by following the practice of never looking at the height of the standard solution in the burette until judgment on the color reaction has been passed, or by working on varying quantities of the substance.

(2) The peculiar character of this sample, which, while it all passed easily through a millimeter sieve, according to the official method, is still, in my opinion, not fine enough. This is largely due to the different nature of the constituents, part being exceedingly pulverulent, while the bone black and potassium nitrate were more granular and tended to rattle out and to a certain extent to separate from the rest of the mass. This was fully understood before the sample was sent out, and hence the special instructions as to careful remixing of this sample.

(3) The very varying strength of the acid and alkali solutions used by the different analysts, as will be shown later.

(4) The too large number of options on reagents to be used in the official Kjeldahl method modified for nitrates as it now stands, and the special liability to nitrogen impurity of some of these reagents.

I am very much of the opinion that zinc and all of its compounds should be excluded from the Kjeldahl and Gunning methods, whether plain or modified, for nitrates. It is by no means necessary to prevent bumping, freshly-heated pumice stone being much better, and in a few cases undoubtedly makes the bumping during distillation begin sooner than it otherwise would do by adding more heavy salts to the solution. As a reagent in the shape of zinc dust, its action is exceedingly violent and causes the violent propulsion of acid and other fumes from the mouth of the flask, entailing danger of mechanical loss, if not of direct loss of nitrogen. This loss is undoubtedly slight, but as results have proved in the past to be somewhat lower by its use, and for this reason zinc sulphid was recommended in its stead, there seems to be little reason for its continuance. Zinc sulphid, it would appear from the results obtained this year, is utterly inadmissible, unless it be used in the form of powdered sphalerite. But even in that case, as in the case of the metallic zinc and zinc dust, it adds unnecessary, heavy, and insoluble zinc salts to the final distillation. The results obtained by the use of sodium thiosulphate do not vary, as in the case where zinc sulphid was used, while they are in every way equal to those obtained by means of zinc dust. In my opinion, the discrepancies that do exist are in no way the fault of the method. In previous years it has always compared well with other reagents in this method, and having the advantage of being a sodium salt, it introduces no new compound into the final solution to complicate its mechanical or chemical action.

The Gunning method modified for nitrates compares very favorably with the Kjeldahl if we eliminate all errors due to impurities in the zinc sulphid. In this method, too, sodium thiosulphate is undoubtedly superior to zinc salts as a reducing agent. The results bear out this statement. The effect of the zinc salts on the solution is here strikingly apparent, for with sodium thiosulphate the solution in the distillation flask remains to the last clear and colorless, while, where zinc sulphid is used, a heavy white precipitate of the hydroxid is thrown down immediately the solution is alkaline. For the same reasons that the Gunning method is superior to the Kjeldahl, so is the Gunning modified for nitrates superior to the Kjeldahl modified, always provided it gives results of equal accuracy, and so far such would seem to be the case.

Sample No. 2—determination of nitric nitrogen.

From—	Analyst.	Ulsch method modified by Street.	Devarda method.	Schultze-Tiemann.
		Per cent.	Per cent.	Per cent.
U. S. Department of Agr., Division of Chemistry.	T. C. Trescot.........	3.18	3.37	....
U. S. Department of Agr., Biochemic Laboratory.	J. A. Emery..........	....	3.46	....
Maryland Exp. Station...............	H. J. Patterson.......	2.87	....	....
New Jersey Exp. Station..............	J. P. Street...........	3.26	....	3.19
Massachusetts Exp. Station..........	C. H. Jones..........	2.21[1]	3.41	....
Connecticut Exp. Station.............	A. W. Ogden.........	2.32[1][2]	....	....
Geneva, N. Y., Exp. Station..........	W. B. Cady...........	3.33	....	....
Geneva, N. Y., Exp. tation..........	A. L. Knisely.........	3.35	....	....
Theoretical............................		3.46	....	....

[1] Average of several unsatisfactory results.   [2] See remarks further on.

The results obtained by the Ulsch method are both good and bad. Four analysts obtain fairly good results, while three send results which are unsatisfactory. Besides these three, six others report varying and unsatisfactory results, but send no figures. One chemist reports that he could obtain no nitrogen by this method. His trouble was undoubtedly due to the fact that 5 grams magnesium oxid, as recommended, is sometimes not sufficient to neutralize the solution, as he stated he followed directions exactly.

The Ulsch-Street method is reported to give excellent results in the New Jersey experiment station, and the result obtained by Mr. Street bears out this statement. Others, however, find trouble, as will be seen by the following remarks and results by Mr. A. W. Ogden:

"Determinations were made on committee sample No. 2, using 1 gram of reduced iron, proceeding strictly according to the directions as given on page 88 of Bulletin No. 35 of the Division of Chemistry, U. S. Department of Agriculture. The results follow:

Sample 2—Determination of nitric nitrogen, Ulsch-Street method.

Determination.	Per cent.
I..........................	2.10
II.........................	1.96
III........................	3.09
IV........................	1.60
V.........................	2.85
VI........................	1.84
VII.......................	3.17
Total	16.61
Average...................	2.37

"The results of seven determinations varied from 1.60 to 3.17 per cent nitrogen as nitrate. On the same sample, using 3 grams of reduced iron, 3.36 was obtained, and using 5 grams of iron, 3.42. Three determinations by Schultze-Tiemann gave 3.18, 3.19, and 3.20.

"The figures obtained by the modified Ulsch method indicate that on a sample containing a high percentage of nitrogen as nitrate, as sample No. 2, 1 gram of

reduced iron is not sufficient to reduce all the nitrate. I was therefore led to try a series of determinations on pure potassium nitrate, using varying qualities of reduced iron. A portion of 0.5 grams of potassium nitrate was used in each case, the portions in each series being aliquots from the same solution. Caustic soda was used instead of magnesia for the distillation.

Series I.

Grams of reduced iron	1	2	3	4	5
Per cent nitrogen obtained	3.28	10.08	13.53	13.58	13.53

Series II.

Grams of reduced iron......	1	1½	2	2½	3	4	5
Per cent nitrogen obtained .	2.31	3.70	4.30	5.50	12.48	13.62	13.56

"It will be seen that the results where less than 3 grams of reduced iron was used are all much too low and that in Series No. 2 the result where 3 grams was used is over 1 per cent low. In all cases where 4 or more grams of reduced iron were used the results are concordant and are reasonably close to the theory. It seems quite probable that 1 gram of reduced iron is sufficient for ordinary fertilizers containing a low percentage of nitrogen as nitrate, as shown by Mr. Street's own results."

The three results obtained by the Devarda method are excellent and would seem to call for a special trial next year. The method is much easier and simpler than that of Ulsch and would appear to be easily adaptable to our ordinary Kjeldahl distillation apparatus.

In using the Devarda method on substances containing organic nitrogen the nitrate should be washed out and made up to a definite volume, and then aliquots may be taken. Organic nitrogen, especially if easily decomposable, is also partially reduced by this method. Equally good results appear to be obtained by the use of the German method, which is identical with the Devarda, except that a mixture of zinc and iron filings is used instead of the Devarda alloy. Mr. Jones obtained 3.44 by this method.

The following additional results were obtained by Mr. Trescot, of the U. S. Department of Agriculture, on pure potassium nitrate.

Absolute method.	Devarda method.	Ulsch method.
Per cent.	*Per cent.*	*Per cent.*
13.83	13.84	13.77
13.82	13.78	13.78
....	13.74	....
....	13.79	....
Average....... 13.83	13.79	13.78
Theory 13.84	....	....

In view of the fact that the Devarda method and that of Kuhn, recently adopted by the German Society of Agriculture Chemists, are so exceedingly simple in manipulation, I would suggest that they be tried by the next reporter. If accurate they would be exceedingly important addition, to our methods, and additions of which we are greatly in need.

With reference to the use of zinc dust instead of potassium sulphid the following results, obtained by Mr. L. A. Vorhees and J. P. Street, in addition to those already noted by Mr. Carpenter, go to show that zinc dust may safely be used:

Number of samples analyzed... 162
Higher than average... 46
Lower than average.. 106
Identical with average 10
Greatest variation from average per cent above.................... .052
Greatest variation from average per cent below.................... .066
A. O. A. C. sample No. 1.................................3.42 and 3.44
A. O. A. C. sample No. 2.................................5.92 and 5.84

Several chemists sent in analyses of their own acid solution by a misunderstanding, and consequently their results are not incorporated, as they are not comparative. Results by all analysts using the silver chlorid method agree closely, as closely, probably, as duplicates by a single analyst might agree even with the greatest care.

Standardization of solutions.

(Reporter's acid.)

Analyst.	By AgCl method.	By H_2SO_4 method.	By tetraoxalate method.
F. W. Woll...................................	....	.003339	.003414
E. B. Voorhees	.003427	....	.003413
C. H. Jones	.003421	....	.003448
H. B. McDonnell	.003401	.003428[1]	.003400[1]
T. L. Lyon	....	.003438	.003430
C. G. Jenter...............................	.003404	.003417	.003403
J. R. Hanahan.............................	....	....	.003440[1][2]
F. W. Morse...............................	.003426	.003495	.003532[2]
L. H. Merrill.............................	.003400	....	....
K. P. McElroy and W. D. Bigelow.............	.003403	.003426	.003331
C. L. Parsons..............................	.003420	.003452	.003406

[1] NaOH used as alkali.
[2] Cochineal used as indicator.

By the H_2SO_4 method the result is what might be expected and the method is not to be recommended. The method requires the ammonia to be titrated against a measured quantity of sulphuric acid, and again against the hydrochloric acid to get the strength of the latter. This makes the volumetric measurements whose errors, due to variation of temperature, calibration, and cleanliness of burettes, etc., might let either offset or multiply each other. All reported good results so far as the standardization of the H_2SO_4 by precipitation with $BaCl_2$ was concerned.

The results by the use of tetraoxalate show, as has long been known, that cochineal can not be used as an indicator. In the hands of some analysts the method gives good results while with others the color reaction is not sharp. The sample sent to Messrs. Bigelow and McElroy was evidently impure, especially as they report titrating it also against NaOH and KOH solutions with the same result. The tetraoxalate was bought in one-ounce bottles, and one of these sent to each analyst. As I stated in my paper before this association last year tetraoxalate that can be depended upon can not be obtained from any firm, but must be made by the chemist himself.

The fact most worthy of note brought out by the results on the standard acid was the great variation in the acid and alkali solutions used by the different chemists of

the country. and I do not believe that good results will ever be reached while this great variation exists. The solutions reported vary as follows:

Strongest ammonia solution reported..............5. 3723 grams to liter.
Weakest ammonia solution reported................1. 109 grams to liter.
Strongest acid solution reported............18. 2285 grams of HCl to liter.
Weakest acid solution reported ....6. 0857 grams of HCl to liter.

Especially on the ammonia solution is this variation apparent. Several chemists of the association state that they have given up the use of ammonia solution altogether on account of the varying character of even a decinormal solution, preferring to use a decinormal NaOH solution, which contains no volatile constituent. The strongest ammonia solution reported could scarcely be smelled with impunity. I believe, also, that the half normal HCl is too strong for accurate work except in the hands of the most experienced chemists, and I believe that they would make more accurate determinations by the use of weaker acid. By the use of our ordinary burettes we can measure but little nearer than one drop, and on some substances a difference of one drop of half normal acid is too wide for accuracy.

RECOMMENDATIONS.

(1) That zinc sulphid be dropped as an agent from all methods of nitrogen determination and sodium thiosulphate be specially recommended in its stead.

(2) That the strength of the official acid solutions shall be stated to be approximately one-fifth normal and the alkali one-tenth normal.

(3) That sulphuric acid standardized by $BaCl_2$, and sodium hydroxid or potassium hydroxid as alkali shall also be official as well as standard hydrochloric acid and ammonia.

(4) That the Gunning method modified for nitrates, as recommended by Mr. Winton in the June, 1892 bulletin of the Connecticut Experimental Station and by Mr. E. B. Voorhees, in his paper before this association last year, be made an official method, with the exception that sodium thiosulphate be the reducing agent used.

(5) That the Gunning method be no longer printed as an alternate method, but be put on a full equality with the Kjeldahl.

THE AVAILABILITY OF NITROGEN IN MIXED FERTILIZERS.

By Dr. Bruno Terne.

At a meeting of station directors and chemists the following schedule[1] of trade values was arranged for use in Connecticut, Massachusetts, Rhode Island, and New Jersey during the season of 1893.

Cents per pound.

Nitrogen of ammonia salts.. 17
Nitrogen in nitrates... 15½
Organic nitrogen in dried and fine-ground fish, meat, and blood, and in mixed fertilizers.. 17½
Organic nitrogen, castor pomace and cotton-seed meal............ 16½
Organic nitrogen, fine ground bone tankage...................... 15
Organic nitrogen, fine medium bone and tankage.................. 9
Organic nitrogen, coarser bone and tankage...................... 7
Organic nitrogen, horn shavings, hair, and coarse fish scrap........ 7

There seems to me a great deal of latitude taken in fixing the valuation of nitrogen in mixed fertilizers. While I readily understand the different valuations adopted by the stations, as a fair representation of the market prices, I fail to understand the reason for alternation of this valuation by mixing any of these products with other fertilizing materials, such as acid phosphates and potash salts. Why, for

[1] Bulletin 93, New Jersey Agricultural Experimental Station.

instance, are horn shavings put down to 7 cents per pound and mixed raised to 17½ cents, while tankage, priced at 9 cents, shall by mixing nearly double its value? If the change of figures is done in the interest of the manufacturer, it is hurtful to the farmer, but it is really not of any benefit to the manufacturer of fertilizers; it is advantageous only to the mixer of acid phosphates with cheap ammoniates, while it works against the use of high-grade ammoniates.

In judging the value of a fertilizer for phosphoric acid, we call for the percentage of phosphoric acid soluble in water or soluble in a solution of citrate of ammonia under the restriction of the official method, without asking for the source of the available phosphoric acid. In passing judgment on the insoluble phosphoric acid the valuation is fixed on the basis of the cheapest source, because it is very difficult to determine the source of the insoluble phosphoric acid in a mixed fertilizer. The same rule applies to potash, with some modification in favor of cotton seed meal mixtures. The same laws should be applied to the determination of ammonia for valuation of mixed fertilizers.

A mixed fertilizer of the same percentage of ammonia, but of different constituents, can have a large and decided variance in the availability of nitrogen contained therein. What are we to understand is available nitrogen? I call available all nitrogen combination soluble in water at 65° F., or practically air temperature.

Unquestionably the nitrogen as a plant food, whether it be in form of an ammonia salt or nitrate, or in the form of any other water soluble combination, is readily absorbed by the roots of the plants and is therefore at once available for the nutrification of the plant. The nitrogenous compounds not soluble in water must pass through the process of fermentation in the soil before they can become available as a plant food. Therefore we are justified in making a distinction between the two forms of combination.

It strikes me that the agricultural chemist should draw the line absolutely in the same way as he does in the case of phosphoric acid. The same principle must be equally right for the nitrogenous compounds. If phosphoric acid in available form is worth three times as much as plant food as insoluble phosphoric acid, the nitrogenous compound soluble in water should, in the same proportion, be worth three times as much as the soluble.

I claim that stations in fixing valuation for mixed fertilizers should make a distinction between (1) available nitrogenous combinations soluble in water at air temperature of 65° F, (2) insoluble nitrogenous compounds.

I grant that the transformation from the water insoluble form to the available form will take place more rapidly than with insoluble phosphoric acid, and would suggest making the difference in valuation one-half instead of one-third. If we adopt the station price for available nitrogenous compounds at 17½ cents, the insoluble should not be higher than 8¾ or 9 cents. In order to prove the correctness of this claim, I have made the following experiments and analyses:

I took samples of fertilizers, products of the markets, which had been previously analyzed, and subjected two grams of each to a careful leaching process on the filter with water with 65° F. until the wash water reached the 1,000 cc. mark. After the filters were dried in the air bath at 100° C. the residuum was carefully taken from the filter and the nitrogen determined with the soda-lime process. I made further mixing of acid phosphate with different ammoniates, previously analyzed, and mixed in each case such proportion as to gain 2 per cent ammonia for the mixing. Samples Nos. 1 and 2 represent a class of complete fertilizers which enter the market in large quantities. Sample No. 3 represents simply an ammoniated phosphate, mostly used in preparation of other goods. Sample No. 4 is a mixture of 1,750 pounds acid rock and 250 pounds azotine of 16 per cent ammonia. Sample No. 5 is a mixture of 1,400 pounds acid rock and 600 pounds high steamed western tankage with 7.03 per cent. Sample No. 6 is a mixture of 1,500 pounds acid rock and 500 pound of cotton-seed meal, yielding 8.07 per cent ammonia.

Estimation and valuation of nitrogen.

Number of sample.	Yield of ammonia.	Yield of ammonia after washing.	Amount of ammonia washed out.	Percentage of water soluble ammonia
	Per cent.	Per cent.	Per cent.	Per cent.
1...................	2. 04	0. 35	1. 69	82. 87
2...................	2. 05	0. 52	1. 53	75. 12
3...................	1. 57	0. 42	1. 15	74. 43
4...................	2. 00	1. 31	0. 69	34. 50
5...................	2. 10	1. 93	0. 17	8. 09
6...................	2. 01	1. 97	0. 04	1. 99

First.		Second.
Valuation for nitrogen per ton, at 17 cents per pound.		Valuation for nitrogen per ton; available nitrogen 17½ cents per pound. Insoluble nitrogen 9 cents per pound.
1.................................	$7. 14	5. 90+0. 62 = $6. 52
2.................................	7. 16	5. 34+0. 92 = 6. 26
3.................................	5. 48	4. 00+0. 74 = 4. 74
4.................................	7. 00	2. 40+2. 34 = 4. 74
5.................................	7. 34	0. 58+3. 46 = 4. 04
6.................................	7. 02	0. 14+3. 54 = 3. 68

These figures do not need much further comment before the Association of Agricultural Chemists. I do not hesitate to say that the figures in column No. 2, give a more true statement of the valuation of nitrogen compounds, as plant food, than those in column No. 1. Practical results have taught the intelligent manufacturer for a long time that the availability of the nitrogen compounds in his products will increase his sale. As the experimental stations are the advisers of the farmers, I think it is a simple claim of justice for the manufacturer that the question of water soluble and insoluble ammonia should be considered by the association and the determination of water soluble ammonia in mixed fertilizers be made a part of the control work of the stations. While samples Nos. 1 and 2 yield 82 per cent, respectively, 75 per cent of the ammonia as water soluble, absolutely in a form ready for assimilation, samples Nos. 5 and 6 offer only 8 per cent, respectively, 2 per cent in the same form. Still, under the present method of official analysis, no notice is taken of this very important point. All brands are adjusted alike. The farmer is left in the dark over the quality of ammonia he is buying, and the manufacturer is not given credit for his work of converting the insoluble nitrogenous compounds into ready plant food. Sample No. 5 is a true representative one of an enormous amount of fertilizers offered in the market. The high steamed tankage, with a yield of 7 to 8 per cent ammonia, contains the latter in a form which necessitates a slow process of fermentation in the soil to make the latter available. From the nature of the process of producing tankage all the easily soluble organic compounds have been extracted in form of the tank water and the parts which resist destruction most are maintained. The mere mixing with an acid phosphate does not alter the quality of the nitrogenous compounds contained therein, and therefore it can not alter its valuation. If the valuation has been fixed at 9 cents per pound, in ordinary tankage, it is a conundrum to me how it can be worth 17½ cents per pound in mixing No. 5.

It is by no means necessary to gain the available ammonia by mixing phosphates with ammonia salts or nitrate of soda. The manufacturer by careful selection of his material and adoption of practical methods is able to convert the organic nitro-

genous compound from the form in which it is most resistive into the most available—the water soluble form—and offers the same in his product in absolute chemical molecular division, which can not be achieved by any mechanical device.

The association has not to deal with the question, how the soluble form of the nitrogenous compounds has been gained, it only deals with results and leaves the solution of ways and means to the producer. As a representative of the manufacturers of fertilizers and as a coworker of your association for a number of years past, I would respectfully ask the association to seriously consider the question of available nitrogenous compounds in mixed fertilizers, and to adopt such methods of nitrogen determination for the control of mixed fertilizers, as shall make a clear distinction between (1) nitrogenous compounds soluble in cold water, and (2) nitrogenous compounds insoluble in cold water. This will enable the stations to scale valuation for mixed fertilizers more nearly to real valuation than the present system will allow.

RESULTS OBTAINED IN STANDARDIZING THE MORE COMMON ACID AND ALKALI SOLUTIONS.[1]

By C. G. JENTER.

The solutions used in this work were the more common standard solutions, viz: two of HCl, one sent out by the reporter, the other a solution used here at the New York Station in our nitrogen work, a solution of H_2SO_4 and one of potassium tetraoxilate. The alkalies used were NaOH, KOH, and NH_4OH. As far as possible a comparison of the three more common indicators, viz: phenolphthalein, litmus and cochineal was also made. The work was done at a temperature of about $22°$C. The solutions were all carefully prepared from pure chemicals and were well preserved.

(a) *HCl.*—This was standardized both gravimetrically and volumetrically. In the former a measured amount of the solution was taken from a burette, a pipette in one case, and run into a weighing bottle and weighed, thus giving the amount of acid taken both by weight and by volume. This was then estimated as AgCl in the ordinary manner.

	HCl in 100 cc.	
	By vol.	By weight.
HCl from the New York Agricultural Experiment Station, taken from a burette and estimated as AgCl	.977961	.978294
	.980834	.980448
	.984064	.978942
	.981030	.979120
Average	.980972	.979201
Reporter's sample 93, taken from a burette	.883623	.884315
	.892777	.885604
	.897691	.886573
	.884132	.885189
Average	.889556	.885420
Reporter's sample, taken from a pipette	.886929	.885627
	.884894	.886774
	.886420	.887219
	.885657	.888411
Average	.885975	.886998

[1] These results were arranged in the form here given, to be incorporated in this report at the request of Prof. C. L. Parsons.

Titration of HCl.

	HCl in 100 cc.	
	By vol.	By weight.
Reporter's sample, taken from a burette and titrated with NH_4OH............	.889156	.890578
	.889156	.886301
	.889156	.886973
Average	.889156	.887951

	Grams in 1 cc. solution.			
	HCl.	NaOH.	KOH.	NH_4OH.
Reporter's HÇl:				
Titrated with NaOH. Phenolphthalein	.008846	.004641	....	....
Litmus....................	.008898	.004619	....	....
Cochineal	.008745	.004697	....	....
KOH. Phenolphthalein..........	.008863	....	.013737	....
Litmus....................	.008866	....	.013918	....
NH_4OH. Litmus....................	.008926	....	....	.003423
Cochineal	.008878	....	....	.003363
N. Y. Station acid:				
Titrated with NaOH. Phenolphthalein	.009798	.004635	....	....
Litmus....................	.009805	.004632	....	....
Cochineal	.009693	.004686	....	....
KOH. Phenolphthalein	.009791	....	.013962	....
Litmus....................	.009828	....	.013919	....
Cochineal	.009883	....	.013897	....
NH_4OH. Litmus....................	.009871	....	....	.003344
Cochineal	.009801	....	....	.003368

(b) H_2SO_4.—The strength of this solution, like that of the HCl, was determined by the gravimetric method (as $BaSO_4$), and by the volumetric method.

	H_2SO_4 in 100 cc.	
	By vol.	By weight.
H_2SO_4, taken from a burette and determined as $BaSO_4$.........................	1.59261	1.58590
	1.60692	1.59050
	1.60271	1.58771
	1.61197	1.60230
Average	1.60330	1.59160

	Grams in 1 cc. solution.			
	H_2SO_4.	NaOH.	KOH.	NH_4OH.
H_2SO_4:				
Titrated with NaOH. Phenolphthalein	.015841	.004673	....	....
Litmus....................	.015896	.004657	....	....
Cochineal	.015671	.004724	....	....
KOH. Phenolphthalein	.015889	....	.014030	....
Litmus....................	.015889	....	.014030	....
Cochineal	.016012	....	.013935	....
NH_4OH. Litmus....................	.016035	....	....	.003356
Cochineal	.015847	....	....	.003395

(c) *Potassium tetraoxalate.* KHC_2O_4, $H_2C_2O_4$, $2H_2O$.—The purity of this salt was determined first by making a standard solution, and second by weighing a portion of the salt and titrating it direct.

In the former case the solution was titrated with the standard alkalies as in ordinary volumetric work. In the latter case approximately 0.2 grams were weighed out, transferred to an Erlenmeyer flask, dissolved in warm water, and after cooling titrated direct with the standard alkalies.

	Grams in 1 cc. solution.			
	Tetraoxa-late.	NaOH.	KOH.	NH$_4$OH.
Tetraoxalate:				
Titrated with NaOH Phenolphthalein..........	.008362	.004635	....	....
Litmus....................	.008372	.004629	....	....
Cochineal	.008127	.004768	....	....
KOH, Phenolphthalein..........	.008426	....	.013854	....
Litmus....................	.008461	....	.013684	....
Cochineal	.008279	....	.014099	....
NH$_4$OH, Litmus....................	.008458	....	....	.003329
Cochineal	.008196	....	....	.003395

	Grams in 1 cc. $\frac{1}{10}$ solution.			
	NaOH (.0040) phenolphth-alein.	KOH (.0056) phenolphth-alein.	NH$_4$OH (.0025) litmus.	NH$_4$OH (.0035) cochi-neal.
Potassium tetraoxalate:				
1. Reporters' sample, 1893................	.003971	.005453	.003456	.003506
	.004003	.005652	.003493	.003463
	.003991	.005614	.003564	.003553
Average	.003992	.005576	.003504	.003507
2. Reporters' sample, 1892, recrystallized 3 times	.004209	.005881	.003627	....
	.004154	.005838	.003618	....
	.004144	.005791	.003627	....
Average	.004169	.005837	.003624	....
3. Reporters' sample, recrystallized 3 times with H$_2$C$_2$O$_4$................·	.604009	.005601	.003484	....
	.003998	.005606	.003483	....
	.003972	.005606	.003487	....
Average..........................	.003993	.005604	.003485	....

The samples of tetraoxalate used were (1) the sample sent out by the nitrogen reporter of this year (1893) and (2 and 3) the sample sent out in 1892. The former, as the result indicates, is practically pure, while the latter gives results which are rather high, even after being recrystallized three times, yet the same samples when recrystallized with H$_2$C$_2$O$_4$ give very good results.

The sample was recrystallized with H$_2$C$_2$O$_4$, the crystals drained and washed with a little water and then dried over H$_2$SO$_4$. The sample was left for over a year without any special precautions, yet at this time it gives the same results that it gave over a year ago.

At 5:30 p. m. the association adjourned to Friday, August 25, 1893, at 9:30 a. m.

SECOND DAY.

MORNING SESSION, FRIDAY.

The convention was called to order by the president at 10 o'clock.

Mr. FREAR moved that a committee on nominations be appointed by the president and directed to report to the convention at 12 o'clock; and that the business meeting of the association be made a special order for that hour.

The motion was carried, and the president appointed as such committee Messrs. Van Slyke, Kedzie, and Winton.

Mr. FREAR stated that a number of reports had been received from reporters who were unable to attend the meeting. He moved that, in order to facilitate business, such reports be referred to a committee of two, who should examine them carefully, in order that they might be presented to the convention in compact form.

The motion was carried, and the president appointed as such committee Messrs. Huston and Kedzie.

Mr. DE ROODE read by title a paper, which he stated should have been presented in connection with his report on phosphoric acid and ought to be incorporated in the proceedings.

There being no objection, the president directed that the paper referred to be printed in the report.

It was so ordered.[1]

Mr. KEDZIE moved the reconsideration of the report on phosphoric acid, together with the recommendations, as adopted at the preceding session. He desired to clear up an ambiguity in the method as it stood in regard to citrate digestion in the bath. So far as he could learn the great majority of agricultural chemists placed the filter and the residue in the solution of ammonium citrate and digested. The method now prescribed directed that the residue be washed off the filter, which was very difficult. Another point he would make was that more uniform results could be obtained by heating the citrate solution to the required temperature in advance, confining the digestion to the half hour. Otherwise some would take from ten to thirty minutes to heat to 65° and carry on the digestion from that point. These variations prevented uniformity of results, which the association aimed at rather than perfect accuracy, which was unattainable.

Mr. MCDONNELL thought it a bad precedent to go back to a matter already settled unless there were very good reasons for so doing. He would be opposed to the introduction of the whole filter.

The PRESIDENT. That is hardly in order. The question is simply shall we reconsider the recommendation.

The motion to reconsider was carried.

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[1] The paper is printed with the discussion on phosphoric acid.—ED.

Mr. KEDZIE moved the adoption of the reporter's recommendation on this subject, as presented at the former session.

Mr. FREAR opposed the motion as covering a modification which had not been generally tried. It was the experience of the association in a majority of cases where methods were adopted which had given good results to a single individual, that objections had arisen which had not at first been apparent. It was not conservative or wise, he thought, to adopt, in an arbitrary method, such as the one under consideration, a modification so far-reaching. He felt that the method as at present worded was open to serious objection. It was beyond question that there was a marked difference in the length of time to which the action of the citrate was extended in different laboratories, owing to the difference in time required to bring the temperature up to 65°. The experiments made by Mr. Williams, and referred to by the speaker yesterday, indicated that the length of time during which the fertilizer was exposed to the action of the citrate was of more importance than a slight variation in temperature. If a fertilizer were exposed in one case four or five minutes longer than in another to a temperature of 63° to 65° there would be a difference in result more marked than that made by a variation in temperature of one or two degrees in the same length of time. If the method was to be modified at all, he thought it would be better to go back to the old wording, and simply insert the flask in a bath at 65°, the bath not to be allowed to fall in temperature more than 2°, and the flask to be kept in the bath for thirty minutes, without regard to the length of time required to bring the temperature back to 65°. More uniform results could be obtained by this method, as had been proved.

Mr. PARSONS was no less opposed to the adoption of anything arbitrary than was Mr. Frear. He understood, however, that the reporter had made a special study of the matter under discussion during the year, had made tests relative to the very point in question, and had found that by the method proposed results as good as or slightly better than those obtained in the other way were had. It had been the practice of a number of chemists in the association to introduce the whole of the filter paper into the citrate solution. In working according to an arbitrary method, such as the present one for citrate soluble, every condition should be made as nearly constant as possible. It was now proposed that the citrate solution be heated to 65° and kept at that point for thirty minutes, and that the phosphate should not be introduced until that temperature had been reached. Now, it was well known that the time required for heating the citrate solution to 65° varied considerably, according to the kind of glass and the thickness of the flask. It would be well to make the conditions under which different chemists worked as nearly alike as possible.

Mr. FREAR agreed entirely with Mr. Parsons in his objection to the present method, but doubted the wisdom of at once adopting a method which had been subjected to test upon only a single class of goods.

Mr. Huston said he would be glad to have the association adopt any method which would increase the chance of uniformity, but he adhered to the opinion he had expressed at the previous session, and considered that the method as it stood would do for the general run of goods. The differences were small. The sample which the reporter had sent out was what might be called a composite sample—a generalized sample, not of such a character as would meet the objections raised in regard to the class of goods presenting the greatest difficulty. During the last two or three years he had said a good deal about the curves of temperature and of time. The temperature curve was greater than the time curve, and any change made would, as he understood, involve both, so that very marked changes in results might be expected in certain goods were the proposed modification made. While he did not believe that in those goods the citrate determinations amounted to much, it was better to have the method as accurate as might be. He did very little work upon goods made from Redonda phosphate. One of the samples sent out by Mr. Lord was made from that, and was perhaps the only sample upon which the association had worked which would meet the point. While no one was more anxious than himself to secure uniformity, he thought the matter should be referred to the reporter for next year for further recommendation. It was better not to adopt a method which would have to be changed in a short time. It was to be remembered that in the present method nothing whatever was said about the temperature in the flask, only that of the bath being prescribed. In the case of some goods it would be radical to change from the temperature of the bath to the temperature of the flask in the bath.

Mr. De Roode was unable to see that the proposed change would alter the conditions otherwise than to afford a greater uniformity, which Mr. Huston would admit was not given by the present method in the class of goods referred to, so far as availability was concerned. If the method gave uniformity of results, it did not matter that it was radical. He claimed that the recommendation, if adopted, would lead to more uniformity in the working conditions.

Mr. Kilgore said that the change proposed would involve the addition of the filter paper with the insoluble residue, and the burning of two filter papers together with the organic material. That was seriously objectionable, because it consumed much time.

Mr. De Roode believed that the loss of time would be more than compensated for by the increased accuracy obtainable by introducing all the fertilizer on the filter paper. In some cases the fertilizer stuck badly, and it was necessary to rub nearly all of the filter paper into the flask.

Mr. McDonnell was opposed to the making of such a change until after further trial. The method had been tried on only one sample, and the results had been but a trifle more uniform than those secured

by the old method. He considered the introduction of the filter paper rather objectionable, as making the washing more difficult. The matter should be referred to the reporter for next year.

Mr. WINTON thought it a mistake to introduce each year some slight change in the method for citrate extraction. The association was now going back to the old method. Some years ago the paper was introduced with the residue. Later, for some reason, that practice had been abandoned, and now it was proposed to readopt it. He had approved the change of temperature from 40° to 65°, but he thought it unwise to make the minor changes from time to time suggested. The term "insoluble phosphoric acid" was given a new meaning at each meeting of the association, and whenever mentioned in print it ought to be provided with an asterisk referring to the year at the foot of the page.

Mr. Kedzie's motion was carried.

Mr. DE ROODE moved that the first recommendation by Mr. Parsons be adopted.

Mr. FREAR saw no objection to the use of zinc blende. It should be noted, however, that it was unsafe to use the manufactured zinc sulphid. He would move to amend the recommendation by adding the words "except introduced in the form of pulverized sphalerite."

Mr. PARSONS said that there was nothing to be gained by the amendment offered. It would necessitate the trouble of pulverizing the blende, which was not likely to be pure, and would have a strong tendency to cause bumping. He saw no reason for complicating the method by adding another reagent. The methods should be as simple as possible.

Mr. FREAR urged, as a general principle, that unless vital error were introduced there might be advantage rather than disadvantage in giving breadth to the method.

Mr. LINDSAY desired to corroborate what Mr. Parsons had said in regard to zinc sulphid. It seemed to be a very treacherous thing to use and had better be omitted.

Mr. FREAR's proposed amendment was not adopted. The recommendation as made by the reporter was agreed to. The second and third recommendations were also agreed to without discussion.

Mr. PARSONS again read his fourth recommendation.

Mr. DE ROODE asked whether, in carrying out the method, it had been found more convenient to add the potassium sulphid later, as Mr. Winton did, than to add all the reagents at once. In his first report Mr. Winton seemed to think that this made some difference in carrying out the method.

Mr. WINTON had given considerable attention to the matter, and in working with pure potassium nitrate he had never been able to secure satisfactory results without shaking the material with a salicylic-acid mixture before adding the reducing agent. His results had never been satisfactory when all the reagents were added at once. Perhaps

this would not introduce any appreciable error in the case of fertilizers containing a low percentage of nitrate, but he thought the matter worthy of consideration. He would be glad to have a method in which all the reagents could be thrown in at the same time.

Mr. PARSONS said it was more convenient to add all at once. There might, however, be danger of a loss of nitrate, especially when zinc dust was used, from the violent reaction which then occurred, but none of the chemists who used thiosulphate had experienced any trouble by reason of putting in all the reagents together. Even in the modified Kjeldahl method which was tried two years ago it was found that zinc dust created a tendency toward loss. He was strongly of opinion that zinc dust should be left out of the method. He would never use it in his own work. It was perfectly safe to use sodium thiosulphate instead of zinc dust, and there would be no loss of nitrogen. He would only ask the association to adopt the modified Gunning method for nitrates, using sodium thiosulphate as the reducing agent.

On motion of Mr. De Roode, the recommendation was adopted.

Mr. PARSONS had not recommended any change in the method of standardizing the acid solution. He believed, however, that it was best to use potassium tetraoxalate. His last recommendation was as follows:

That the Gunning method be no longer printed as an alternate method, but be put on a full equality with the Kjeldahl method.

On page 200 of last year's bulletin there appears the following:

For ordinary work, half normal acid—i. e., acid containing 18.2285 grams of hydrochloric acid to the liter—is recommended; for work in determining very small amounts of nitrogen, one-tenth normal acid is recommended.

This method was only recommended, not required, and hence the widely different results. He recommended that the wording at the top of page 200 be made "standard acid, approximately one-fifth normal, which has been determined by silver chlorid," and that further on the wording be so changed as to require the use of acid one-tenth normal.

Mr. ROSS. The point suggested by the reporter a few moments ago, in regard to the variability of even tenth normal ammonia under changes of temperature, called to my mind the experience we have had in Louisiana as to the employment of ammonia for the standard alkali. With every possible precaution we have found it impossible to maintain an ammonia solution of anything like constant strength, and have been obliged to use sodium hydrate or potassium hydrate as the standard alkali. We have also experienced the same difficulty with hydrochloric acid. Considering the fact that the acid is somewhat more concentrated than the ammonia solution, the loss by the escape of hydrochloric acid as gas has also tended to impair the accuracy of our results, so that we have been forced to use sulphuric acid and potassium or sodium hydrate in order to obtain anything like accurate results, and in order

to secure solutions which could be maintained permanently at something approaching a constant strength. I should be glad if we were not confined strictly to one acid or one alkali as long as this very great difficulty is experienced, especially where the climate is so warm as it is in our section of the country during so considerable a portion of the year.

Mr. PAYNE lived in a moderate climate, and at one time experienced a good deal of trouble with ammonium. He had found relief by storing the solution in bottles sealed with paraffin. He made about two gallons at a time, and tests showed that the solution would hold its strength indefinitely.

Mr. McDONNELL had abandoned the use of hydrochloric acid and ammonium, and now used sodium hydrate and sulphuric acid. Sodium hydrate retained its strength much better than did ammonium, and he saw no reason why some discretion should not be allowed in the matter of acids and alkalis. He would like to have the use of sulphuric acid and sodium hydrate permitted as an alternate method, and would make a motion to that effect.

Mr. PARSONS. My recommendation is simply that our acid solution be stated as one-fifth normal and our ammonia solution as one-tenth normal. I should be very glad to see a little option left as to the acid and alkali used.

Mr. HARTWELL. Do you mean one-fifth or one-half?

Mr. PARSONS. I mean one-fifth.

Mr. VAN SLYKE thought it would be well to say "one-fifth normal is recommended." He would propose that as an amendment.

Mr. DE ROODE said that the amendment would not accomplish the purpose of the reporter, which was a good one. One-tenth normal hydrochloric acid was very treacherous. Tests had shown that it might change after remaining constant for six months. He thought it would be a good thing to make the use of one-fifth normal acid compulsory, not allowing so much latitude to the analyst. The cost of a new overflow pipe or burette was inconsiderable.

Mr. HUSTON thought it worthy of note that the use of fifth-normal acid and alkali would tend to the reduction in amount of material taken for estimation. He believed that in all the analytical work done by the association larger samples should be taken. While the work should be conducted with all due regard to the final results, there should be a reasonable ratio between accuracy of the first and last parts of the operation. He did not suppose that the adoption of fifth-normal acid would result in any inconvenience to the majority, but he would prefer to have the direction remain as not stronger than one-half normal, and one-fifth normal recommended.

Mr. WINTON understood that it had not been customary to adopt changes in methods until after submission to the analysts for experi-

ment. He did not think that the proposed change had been a matter of study during the past year.

Mr. PARSONS was of opinion that it had.

Mr. WINTON inquired whether the question as to using one-half normal or one-tenth normal acid had been submitted to the chemists.

Mr. PARSONS replied that it had not, as most of the members of the association already employed acid of one-fifth normal strength.

Mr. WINTON said that in Connecticut they had used the one-half normal, with burettes of small caliber to correspond. Their work was more accurate when the strong solution was used in the accurately calibrated burette of small diameter. The question should be submitted to the association for experiment before a change was made.

The PRESIDENT. The motion is upon the amendment proposed by Mr. Van Slyke, recommending the use of one-fifth normal acid.

The motion was lost.

The PRESIDENT. The vote is now upon the original recommendation as made by the reporter.

Mr. PARSONS. I do not think that my recommendation will be adopted, but I shall stick to it, and I wish that it be understood. It is that the acid solution shall be approximately one-fifth normal and the alkali solution approximately one-tenth normal.

The recommendation was not adopted.

Mr. WINTON used half-normal acid, but did not think that others should be compelled to do so. When one was accustomed to a particular strength of solution he should be allowed to use it.

Mr. FREAR moved that the acid and alkali used in the direct titration be left to the option of the chemist.

The motion was carried.

Mr. PARSONS. I move that where H_2SO_4 is used as the standard the strength of the acid be determined by means of precipitation as $BaSO_4$.

The motion was carried.

The recommendations, as amended, were adopted as a whole.

The President called for the report on potash. In the absence of the reporter the paper was read by Mr. Huston.

REPORT ON POTASH.

By NORMAN ROBINSON.

Your reporter on potash begs leave to submit the following as the result of his work for the current year.

For reasons which it is not necessary now to recapitulate, but which were fully explained in a circular issued March 24, it was found impracticable to send out samples at an earlier date. However, notwithstanding the lateness of the season, over forty samples were distributed to chemists in this country and in Europe, who had signified their willingness to take part in the work. It was early decided to make two original samples. The first consisted of an ordinary fertilizer compounded for the Florida market by one of our best manufacturers, and containing bone, dried blood, etc., with a liberal supply of potash, mostly in the form of sulphate, and pre-

senting the usual analytical difficulties. The second was of mixed potash salts, sulphate, "muriate," "double salt," "kainit," etc., with about 5 per cent of the difficultly soluble triple sulphate of potassium, calcium, and magnesium (polyhalite), a compound often present in kainit, and which is not infrequently found in small proportions in other German potash salts. This was added to fairly test the question of the accurate estimation of potash by the official method when "polyhalite" is present in the proportions indicated.

In making up these samples, no pains were spared to render the mixture as uniform as possible. The sample in each case was ground so that it would all readily pass through a "20-mesh" sieve. It was then passed through this sieve ten times in succession, with careful and protracted hand mixing between each sifting. It was then reground until it would all readily go through a "40-mesh" sieve, and then passed successively twenty times through this, with careful hand mixing, as before. At the conclusion of the work, two samples of half a gram each were taken from opposite sides of the pile of "fertilizer," and two samples of one-fourth gram each from the "mixed potash." These four samples were then analyzed "in parallel;" and as the corresponding samples gave substantially identical results, it was thought safe to assume that the products in each case were as nearly uniform in composition as it was possible to make them.

As German and other authorities entitled to the highest respect have recently challenged the accuracy of the Lindo-Gladding method of potash estimation, it was recommended that the work of the association for the present year should be especially directed to an investigation of real or supposed sources of error in this official method. As the presence of sulphuric acid, free and combined, has been held responsible for a considerable share of the assumed errors, it was suggested that the potash in the fertilizer sample be determined both by the Lindo-Gladding and the "alternate" method. It was thought that a tabulated series of such estimates, with the sulphuric acid present in the one case and absent in the other, might throw some light upon the question at issue.

In the case of the "mixed potash salts," it was suggested that the potash be determined both by the official method, as described in paragraph 3, page 197, in the last Report of the Association, Bulletin 35, and also by the Anhalt or Stassfurt method, as described in paragraph 1, page 63, of the same report. Something like a fair comparative estimate of the respective values of these methods it was believed could thus be secured.

Original work was also urged in discovering and pointing out any sources of error that either of these methods might involve, and the contrivance of any new methods, tending to ease and simplicity of manipulation or greater accuracy in results.

A few words may not be inappropriate in regard to the "new departure" which has this year been successfully carried out in securing the coöperation of prominent German chemists in the work of the association. The writer believes that the official agricultural chemists of this country are to be sincerely congratulated on the auspicious beginning which has thus been made in international and coöperative work—a work which if followed up to its logical results can hardly fail to have a very important and wide reaching influence upon the perfection of methods in agricultural chemistry, both in this country and in Europe.

Very early in the year I addressed a letter to the celebrated German chemist, Dr. Max Maercker, privy counselor, director of the experiment station at Halle, director of the Agricultural Central Association of the Province of Saxony, and president of the German association of official chemists, cordially inviting him in the name of the Association of Official Agricultural Chemists to take part with us in the work of the current year. To this letter a prompt reply was received of which the following is a translation:

AGRICULTURAL AND CHEMICAL EXPERIMENT STATION,
Halle on the Saale, Germany, January 17, 1893.

Mr. NORMAN ROBINSON,
   State Chemist of Florida, United States of America:

In accordance with the suggestion contained in your letter of December 28, I will gladly undertake to have the manure commission of the "Association of Agricultural Experiment Stations" in the German Empire analyze any samples of potash salts you may send me; and I request that such samples be sent to my address, 10 Karl street, Halle, Germany. I will then immediately transmit the samples to my colleagues of the manure commission. As this commission consists of six members, I request that you send six packages of each sample of potash salts.

The "Association of Experiment Stations" acting in conjunction with the Stassfort manufacturers of potash, has recently simplified and perfected the method of determining potash. You can easily obtain from our friend von Herff the directions for making the analysis. The "Association of Experiment Stations" will employ this method in the analysis of your samples.

Most respectfully yours,

MAERCKER.

In accordance with these suggestions, samples were transmitted as directed, through the politeness of Dr. B. von Herff, of the German Kali Syndicate, whose kind offices in this and other matters I wish especially to acknowledge, and to whose efficient aid much of our success in securing the coöperation of our German brethren is doubtless due.

Subsequently samples were sent, as they were requested, to other chemists in Germany who became interested in the work. As the interest in the matter seemed at first to be very general, and something like thirty chemists in this country expressed a willingness to assist, I had hoped that this year we should have an unusually long and valuable list of contributors and contributions to this potash work. Of all those to whom samples were sent, barely eight have reported. Of course, this labor is purely gratuitous, and no man is under other than scientific obligations to undertake it. Our German brethren have made a very different record. Every chemist that received samples did the work, and reported promptly. Comment is unnecessary. There is also a very close approximation in the results given by our German brethren which is not so observable in the work done in this country.

Determination of potash by the Lindo-Gladding and "Alternate," and by the Stassfurt and other German methods.

AMERICAN CHEMISTS.

Experiment station, college, etc.	Analyst.	Sample No. 1.		Sample No. 2.	
		Per cent of K_2O Lindo-Gladd.	Per cent of K_2O Alternate.	Per cent of K_2O Lindo-Gladd.	Per cent of K_2O Stassfurt.
Michigan Agricultural College.....	W. L. Rossman, average of 4 est.	12.51	12.77	37.33	36.825
North Carolina Ag. Ex. Station	R. E. Noble, average of 2 est.	13.22	13.175	38.32	38.30
North Carolina Ag. Ex. Station	F. B. Carpenter, average of 2 est.	13.08	13.13	38.32	....
New Hampshire Ag. Ex. Station ...	F.W.Moore and F. D.Fuller.	12.13	12.38	37.00	....
Maine State Ag. College.............	J. M. Bartlett	12.94	12.76	38.64	38.22
Maine State Ag. College.............	J. M. Bartlett	12.88	12.78	38.64	38.32
New Jersey Ag. Ex. Station	Louis A. Voorhees..........	12.76	....	38.04	....
School of Mines, Columbia College .	Elwyn Waller..............	13.10	....	38.50	37.621
School of Mines, Columbia College .	Elwyn Waller	12.944	....	38.78	37.467
School of Mines, Columbia College .	Elwyn Waller	....	....	....	37.592
Average......................		12.84	12.775	38.38	37.692

Determination of potash by the Lindo-Gladding, etc.—Continued.

EUROPEAN CHEMISTS.

Experiment station, etc.	Analyst.	Per cent of K_2O by German methods.[1]	Per cent of K_2O by Stassfurt method.[2]
Experiment station at Darmstadt	Dr. Wagner	13.34	37.96
Experiment station at Halle		13.10	37.89
Experiment station at Moeckern	Dr. Koehler	13.44	37.81
Experiment station at Moeckern	Dr. Koehler	13.34	37.81
Experiment station at Hildersheim	Dr. Berendes	13.19	38.14
Directors of Anhalt Salt Works	Dr. Zimmerman	13.23	38.06
Verkaufs Syndicate, Stassfurt	Dr. Tietjens	13.20	37.80
Royal Prussian mines	Dr. Berendes	13.13	37.88
Consolidated Alkali Works, Westeregeln	Drs. Wagner and R. Caspari	13.25	37.98
Consolidated Alkali Works, Westeregeln	Drs. Wagner and R. Caspari, Perchlorate Method	13.09	37.82
Average		13.23	37.91

[1] Sample No. 1.   [2] Sample No. 2.

EXTRACTS FROM CORRESPONDENCE.

AMERICAN CHEMISTS.

Dr. Elwyn Waller, of the School of Mines, Columbia College, after giving a very interesting and detailed account of his mode of procedure with the Stassfurt method, describes some experiments, that are quite in line with my own conclusions, which will be subsequently given, and which I, therefore, quote in full. He says:

"The idea naturally occurred to me that the Stassfurt method might possibly yield low results on account of the vexatious peculiarity of $BaSO_4$ (dragging down other salts with it, especially those of potash). With a view to testing this point, some pure potash alum was taken from the same lot on which I obtained by Lindo-Gladding method, with NaCl 9.816 per cent; without, 9.836 per cent. Theoretical 9.90 per cent. Twenty-five grams were dissolved by fifteen minutes' boiling with 100 cc. water and 1.5 cc. concentrated HCl. This solution was poured into a 250 cc. flask containing 100 cc. cold saturated $CaSO_4$ solution, rinsed in, and after cooling diluted to the mark. After mixing well, 100 cc. were filtered off, placed in the 500 cc. flask, brought to boiling and $BaCl_2$ solution (containing 52 grams in 500 cc.) added from a burette as before (104 cc. were used, which proved this time to have afforded a slight excess of $BaCl_2$). After cooling, the solution was diluted to 502.5 cc. and 50 cc. lots filtered off for precipitation by H_2PtCl_6. The results were:

	K_2PtCl_6	K_2O.
	Gram.	*Per cent.*
Alum Stassfurt method 3 cc. H_2PtCl_6	0.4834	9.334
Alum Stassfurt method 3 cc. H_2PtCl_6	0.4826	9.318

"My conclusions would be: The Stassfurt method has the advantage of averaging results on a larger quantity of material. The precipitate obtained is always more presentable. These advantages are more than offset on the score of accuracy by serious defects. The method is much more elaborate and less easy of execution than the Lindo-Gladding.

"The method seems always to give low results on sulphate, apparently because the the BaSO₄ precipitated carries potassium salts out of the solution. Working with the Stassfurt method as one ordinarily must, the adjustment of the exact proportion of BaCl₂ solution to be added is not easily accomplished; if too little BaCl₂ is added the precipitate of K₂PtCl₆ contains sulphates insoluble in alcohol; if too much BaCl₂ is added, the precipitate will contain BaCl₂, also insoluble in alcohol. Cases may perhaps occur where one or the other of these errors will counterbalance the error due to loss of potassium salt with the BaSO₄, and a correct result may be obtained, but the method cannot be commended on that account. The Lindo-Gladding method, although no doubt capable of improvement, is inherently more accurate than the Stassfurt method."

F. W. Morse, of the New Hampshire Station, writes:

For sample (1): "The alternate method was modified by using the same amount of water and the same length of time for boiling as in the Lindo-Gladding method. Is it not inconsistent to have such difference in quantities of water and length of time?"

In estimating sample (2) mixed potash salts: "The alternate method was modified by using the strength of HCl (2 per cent solution), and removing only H₂SO₄ by BaCl₂, then removing excess of Ba by (NH₄)₂CO₃. Results, 37.19 per cent."

W. L. Rossman, Michigan Agricultural College, says: "The Lindo-Gladding method was strictly followed, except that 5 grams were boiled with 150 water, and made up to 250 cc. Sample No. 2, by German method by two estimates, gave 36.80 and 36.85 per cent."

R. K. Noble, of the North Carolina Experiment Station, writes: "In the Anhalt method correction for the ppt. of BaSO₄ was made as recommended. The results agree very closely with the Lindo-Gladding method. It is considerable trouble to precipitate all the SO₃ without an excess of BaCl₂, and for this reason is more troublesome than the Lindo-Gladding method. In a second case when the Anhalt method was used and no correction made, the result was within the limits of error, but the result was 12 per cent higher than with the correction."

Some additional work, undertaken partly at my request and just reported, is of special interest. Mr. Noble finds, in the case of the mixed potash salts, first, that the pure water dissolved slightly less than water with 2 per cent HCl added, but that in practice, pure water is a sufficiently reliable solvent; second, that the (NH₄)C₂O₄ precipitate holds potash only recoverable by resolution in HCl; third, that he does not find any additional potash taken up by the NH₄Cl solution. On the other hand, he is able to recover somewhat less after using it to wash the KPtCl₆ precipitate than before.

Your reporter has had a great many similar experiences.

GERMAN CHEMISTS.

As the same circular was forwarded to Europe, with the samples, that was sent to chemists in this country, it was hoped, by the suggested mutual interchange, trial, and comparison of methods, that valuable data might be obtained. Our German friends either did not receive this circular, or did not deem it wise to carry out its suggestions. Nevertheless, some very interesting and important facts are brought out in the appended extracts from our German correspondence. For the excellent subjoined translation of this correspondence, I have again to acknowledge my indebtedness to Dr. B. von Herff.

From the Consolidated Alkali Works, at Westeregeln, under date of May 16, 1893, and reporting to Dr. Tietjens and E. Lierke, Laboratory of the Sale Syndicate, Leopoldshall, Drs. Wagner and R. Caspari say:

"Herewith we beg to communicate to you the results of our investigations of the American fertilizer samples. In the course of our work the fact was again clearly and distinctly brought out that the improved perchlorate method is decidedly

superior to the old platinum chlorid method as regards rapid execution and simple manipulation. This superiority is especially perceptible in the analyses of mixed fertilizers, containing phosphoric acid. This, for the reason that the laborious and time-taking separation of the alkalies, which is necessary in the platinum chlorid method, in order to get rid of the phosphoric acid, is entirely avoided. We employed the platinum chlorid method with sample No. 1, mixed German potash salts, according to method No. 3 of the syndicate proceedings of August 9, 1892.

Sample No. 2, fertilizer: The presence of nitrogen and phosphoric acid in the sample was indicated by Dr. Robinson and confirmed by preliminary examination. This necessitated the employment of the complicated method with the separation of the alkalies, which, however, was simplified by us taking smaller aliquot portions of the liquids after each precipitation and filtration. Ten grams of the substance were ignited in a porcelain crucible at low red heat, dissolved by boiling in a 500 cc. flask with 300 cc. water and 20 cc. of 25 per cent of hydrochloric acid. Sulphuric acid was precipitated in the boiling liquid by acidulated barium chlorid solution after the well-known method as agreed upon by the syndicate (meeting of August 9, 1892), 25 cc. of the filtrate were evaporated almost to dryness upon the water bath with an excess of chlorid of iron. The residue was taken up with water and milk of lime was added until the liquid reacted alkaline. The mixture was then made up to 500 cc. and to 200 cc. of the filtered solution, ammonium carbonate, a little ammonium oxalate and a little ammonia were added in a 5-liter flask. A heating upon the water bath for a short while favors the formation of a granulated precipitate, which readily settles. The liquid was filled up to the mark, and after standing about two hours was filtered. One hundred cc. of the filtrate were evaporated to dryness upon the water bath in a platinum dish. The ammonia salts were driven off by heating. The residue was taken up with water, filtered, washed, acidulated with hydrochloric acid and treated in the well-known manner with platinum chlorid.

Perchlorate method.

(1) Sample of mixed potash salts:

Five grams of the substance were freed from sulphuric acid in a 500 cc. flask, according to the method described in Ztschr. f. angew. Chem., 1893, p. 73, and 0.4 grams of the substance were treated after the method described in Ztschr. f. angew. Chem., 1892, 8, and 1891, 23, p. 691. The filtration was made through a weighed asbestos tube.

(2) Fertilizer:

In view of the possibility that the perchloric acid might act upon and transform the nitrogen compounds of the sample into ammonia salts, 10 grams of the substance were ignited in a porcelain crucible to a low red heat and then boiled in a 500 cc. flask with 300 cc. water and 20 cc. concentrated hydrochloric acid, and precipitated with acidulated barium chlorid solution; 40 cc. = 0.8-grams substance, were then treated according to the method described in Ztschr. f. angew. Chem., 1893, 3, 71. The whole work was performed within a few hours, and with very little trouble, while the employment of the platinum method would have consumed at least four times the amount of time.

Results (closely agreeing with duplicate analyses):

	Mixed German potash salts.	Fertilizer.
	Per cent.	*Per cent.*
Platinum method	37. 98 K_2O	13. 25 K_2O
Perchlorate method.....................	37. 82 K_2O	13. 09 K_2O

In his report to Chief Director Maercker, under date of May 30, 1893, Dr. Berendes, of Stassfurt, says:

The samples sent us for analyses by Dr. Robinson, of Florida, gave the following results:

(1) Mixed fertilizer (sulphate, dried blood, bone meal) 13.13 per cent potash.

(2) Mixture of various potash salts, 37.88 per cent potash.

The method employed for sample No. 2 was one of the methods agreed upon by the German potash industry, described under the heading A 2. The analysis of sample No. 1 was made as follows: Ten grams substance were ignited in a platinum dish, the residue was dissolved with about 350 cc. water and 15 cc. hydochloric acid in a 500 cc. flask and the sulphuric acid precipitated in the usual manner, with chlorid of barium. The liquid, after cooling, was filled up to the mark, mixed and filtered. 250 cc. of the filtrate were evaporated almost to dryness with an excess of chlorid of iron. The residue was taken up with hot water and poured in a 500 cc. flask. Lime water was then added until the liquid reacted alkaline. After filling up to the mark, filtering etc., 250 cc. of the filtrate were treated with ammonium carbonate, ammonia, and a little oxalate of ammonia, and heated in a 500 cc. flask. After filling up to the mark, filtering, etc., 100 cc. were evaporated in a platinum dish. The residue was ignited and the determination of potash made in the usual manner.

Another determination was made without removing the phosphoric acid. Ten grams of the substance were ignited. The sulphuric acid was precipitated in a 500 cc. flask. 25 cc. of the liquid were evaporated directly with platinum chlorid. The analysis gave 13.27 per cent potash.

On motion, the report was accepted.

Note.—From a previously dissolved sample of the mixed German potash salts sent out by the reporter, 200 cc. was taken, corresponding to 5 grams of material, precipitated as accurately as possible with BaCl₂ and made after cooling to 251.25 cc., and after vigorous shaking set aside for a few hours to secure the complete subsidence of the $BaSO_4$. (The volume of the latter was determined at 1.25 cc. by calculation from the $BaCl_2$ used and confirmed by subsequent weighing.) Ten cc. accurately measured of the clear solution corresponding to one-fifth gram gave 0.392 of $K_2PtCl_6 \times 5 = 1.960$ or 37.84 per cent, which closely approximate the average of the estimates made by German chemists, as much of the remaining liquid as could be moved without disturbing the precipitate was now poured off into a clean, dry beaker, and the precipitate with portions of this same solution was washed into an accurately calibrated 50 cc. measuring cylinder and again allowed to settle. After three hours the precipitate with the mingled solution occupied a volume of 16 cc. and did not seem inclined to go any lower. The clear liquid was now taken out with a pipette until exactly 21.25 cc. remained. The whole was now poured upon a dry 9 cm. Schleicher and Schüll filter and exactly 10 cc. filtered off and rejected. This of course left 10 cc. of the solution and 1.25 cc. of the $BaSO_4$. The filter was repeatedly washed with hot water until no reaction was observable with argentic nitrate. This last 10 cc. of solution with the washings gave .4276 of K_2PtCl_6 or 22.6 milligrams more than the first 10 cc. of solution examined, showing that this amount of potash was held up or occluded by the $BaSO_4$ in a form that boiling water, would remove. The filter with its precipitate was then boiled twice in 25 cc. dilute HCl (1 to 5). In this way 44 milligrams more K_2PtCl_6 were obtained. The total amount of K_2PtCl_6 recovered from the $BaSO_4$ precipitate was 66.6 milligrams or 0.01288 to each gram of substance = 0.247 per cent or nearly one-fourth of one per cent. Other tests of the same material in smaller quantity gave considerably higher results, but as great care was taken to secure accuracy in every stage of the process, it is believed that the above fairly indicates the average loss where this particular material is estimated by the Stassfurt method. Much more extended investigations are required before any confident statements can be made as to the average loss from occluded potash when this method is employed.

It was moved that the Stassfurt method be referred to the reporter on potash for next year, with direction to make a recommendation thereon.

The motion was carried.

ELECTION OF OFFICERS.

Mr. Van Slyke, for his committee on officers for next year, reported the following nominations:

For president, Mr. E. B. Voorhees.

For vice-president, Mr. H. A. Huston.

For secretary, Mr. H. W. Wiley.

For members of executive committee, Messrs. B. B. Ross and Charles D. Woods.

On motion, the stenographer (the secretary being absent) was instructed to cast the ballot of the association for the entire list of nominees. He did so, and the gentlemen named were declared elected.

MISCELLANEOUS BUSINESS.

Mr. HUSTON desired to offer a suggestion, perhaps not properly to be embodied in the form of a motion. He referred to the relations existing between official and commercial chemists. Marked differences had been found between the results obtained by the two classes of chemists on the same samples, worked by the same official methods. In such cases the commercial chemists were not always able to assure themselves that the samples were identical, owing to their inability to obtain a part of the material after the official analysis had been made. Some action which would promote uniformity in this matter would be productive of good feeling on both sides.

Mr. PAYNE said that in some States the commissioners made it a rule to allow no sample to leave their laboratories for further analysis.

Mr. FREAR saw no objection to giving a portion of the official sample to a chemist representing a manufacturer. He always retained a portion for use in further tests should they be required.

Mr. GAINES moved that a committee be appointed to consider the subject and report a plan for the settlement of the difficulty suggested.

Mr. VAN SLYKE objected that the matter was outside of the association's work, which was the discussion of methods of analysis.

The motion was lost.

Mr. FREAR. I think before we adjourn we ought to express our appreciation of the favors we have received from the Secretary of Agriculture and from the officers of the World's Fair Congress Auxiliary. I move the following resolutions:

Resolved, That the thanks of this association be tendered the honorable Secretary of Agriculture for the favors granted by him in detailing Mr. Talma Drew to report the proceedings of the tenth annual meeting, and for his promise to publish these proceedings as a bulletin of the Division of Chemistry.

Resolved, That the thanks of the association be tendered also to the officers of the World's Congress Auxiliary for the use of the Art Memorial Institute for the present meeting.

The motion was carried unanimously.

A recess was then taken.

AFTERNOON SESSION, FRIDAY.

The association was called to order by the president at 1:30 p. m.

The president called for the report on soil analysis. In the absence of Mr. Hilgard, Mr. Huston read the following paper:

REPORT ON THE METHODS OF PHYSICAL AND CHEMICAL SOIL ANALYSIS.

By E. W. HILGARD.

Before entering upon the details of physical and chemical soil investigation, it may not be superfluous to define what are the objects to be accomplished in such investigations, both from the standpoint of the farmer and of the agricultural expert.

It is well understood that in the present state of knowledge, the chemical analysis of soils long cultivated and fertilized affords, alone, but a limited amount of information regarding their productiveness, although always useful in defining their general character and obvious deficiencies. In the case of those cultivated without fertilization, even for a considerable length of time, chemical analysis will afford us valuable indications not only regarding their general character, but also in respect to the important soil ingredients most likely to have been reduced below the level of profitable culture. Yet, so long as virgin spots fairly representing cultivated lands exist, the results obtained by the examination of the former can be fruitfully applied to the latter.

In virgin soils the indications, as experience has shown, become so definite as to permit a close forecasting not only of the general character and value of lands not yet brought under cultivation, but also of their best adapations and of the probable duration of productiveness without fertilization. Chemical analysis can thus, when combined with the physical, geological, and botanical examination of soils, give information of such direct practical importance that in the newer States and in the Territories it becomes a reliable guide to the settler in the choice of lands and cultures. Such work is therefore peculiarly the province of the experiment stations west of the Mississippi River.

As regards physical soil analysis, as practiced in Europe, its results have fallen far short of the expectations which, theoretically, it might be expected to fulfill, viz, the definite measurement of the " tilling qualities " and of other physical coefficients of soils. The judgment regarding these points deducible from the great majority of mechanical soil analysis as usually made and published, affords hardly more information than could be derived from mere hand tests on the dry and wet soil. Yet it is hardly doubtful that a proper study of the physical composition can be made to yield a much deeper insight into the functions of the several physical soil ingredients and, consequently, into the means of modifying the physical properties to the best advantage.

One important function of such examinations (about the feasibility of which there can be no reasonable question) is the identification of uncultivated soils with

those of lands already under cultivation, thus permitting the application to new lands of experience already acquired. But in order to attain such results, not only must the mode of sampling in the field, the preparation of the samples for analysis, and the physical and chemical analysis itself, be conducted upon a well-considered and uniform system, but the conditions of occurrence, the "lay," depth, climate, natural vegetation, etc., must be known as fully as possible, since all these factors must be taken into consideration in interpreting, for practical purposes, the results of the physical and chemical examination. Moreover, in determining the conditions to be fulfilled and the *modus operandi*, all arbitrary conventions should be avoided as much as possible, since all such are sure to be violated, sooner or later, by workers who consider themselves as much entitled to exercise of judgment as anyone else. In developing the methods originally suggested by D. D. Owen, I have therefore sought to establish a rational basis inherent in the nature of the case, as far as possible, and to determine maxima or minima rather than arbitrarily assumed means. The detailed motivation of all these points would increase this report far beyond the admissible limits; but references are given to the publications in which such points are discussed.

I have also, for brevity's sake, omitted a detailed description of the manipulations in the case of well-known analytical methods, mentioning only some critical points upon which success depends, but taking for granted the following out of the accepted precepts.

The Sampling of Soils.

Since the practical utility of soil work would be greatly impaired were it dependent only upon the personal exploration of the wide domain by the experiment station officers, I have formulated, and long used successfully, the following "Directions for taking Soil Samples," that are forwarded either to persons desiring information as to their lands, or to intelligent farmers residing in regions of which the soils are to be investigated. It is often surprising how accurate and graphic are the data so obtained.

In taking soil specimens for examination the following directions should be carefully observed, always bearing in mind that the analysis of a soil is a long and tedious operation, which can not be indefinitely repeated:

(1) Do not take samples indiscriminately from any locality you may chance to be interested in, but consider what are the two or three chief varieties of soil which, with their intermixtures, make up the cultivatable area of your region, and carefully sample these first of all; then sample your particular soil with reference to these typical ones.

(2) As a rule, and whenever possible, take specimens only from spots that have not been cultivated, nor are otherwise likely to have been changed from their original condition of "virgin soils"—*e. g.*, not from ground frequently trodden over, such as roadsides, cattle paths, or small pastures, squirrel holes, stumps, or even at the foot of trees, or spots that have been washed by rains or streams, so as to have experienced a noticeable change, and not to be a fair representative of their kind.

(3) Observe and record carefully the normal vegetation, trees, herbs, grass, etc., of the average land; avoid spots showing unusual growth, whether in kind or quality, as such are likely to have received some animal manure or other outside addition.

(4) Always take specimens from more than one spot judged to be a fair representative of the soil intended to be examined as an additional guarantee of a fair average.

(5) After selecting a proper spot pull up the plants growing on it, and scrape off the surface lightly with a sharp tool to remove half-decayed vegetable matter not forming part of the soil as yet. Dig a vertical hole, like a post hole, at least 20 inches deep. Scrape the sides clean so as to see at what depth the change of tint

occurs which marks the downward limit of the surface soil, and record it. Take at least half a bushel of the earth above this limit, and on a cloth (jute bagging should not be used for this purpose, as its fibers, etc., become intermixed with the soil) or paper break it up and mix thoroughly, and put up at least a pint of it in a sack or package for examination. This specimen will, ordinarily, constitute the "soil." Should the change of color occur at a less depth than 6 inches the fact should be noted, but the specimen taken to that depth nevertheless, since it is the least to which rational culture can be supposed to reach.

In case the difference in the character of a shallow surface-soil and its subsoil should be unusually great—as may be the case in tule or other alluvial lands or in rocky districts—a separate example of that surface soil should be taken, besides the one to the depth of 6 inches.

Specimens of salty or "alkali" soils should, as a rule, be taken only toward the end of the dry season, when they will contain the maximum amount of the injurious ingredients which it may be necessary to neutralize.

(6) Whatever lies beneath the line of change, or below the minimum depth of 6 inches, will constitute the "subsoil." But should the change of color occur at a greater depth than 12 inches the "soil" specimen should nevertheless be taken to the depth of 12 inches only, which is the limit of ordinary tillage; then another specimen from that depth down to the line of change, and then the subsoil specimen beneath that line.

The depth down to which the last should be taken will depend on circumstances. It is always necessary to know what constitutes the foundation of a soil, down to the depth of 3 feet at least, since the question of drainage, resistance to drought, etc., will depend essentially upon the nature of the substratum. But in ordinary cases 10 or 12 inches of subsoil will be sufficient for the purposes of examination in the laboratory. The specimen should be taken in other respects precisely like that of the surface soil, while that of the material underlying this "subsoil" may be taken with less exactness, perhaps at some ditch or other easily accessible point, and should not be broken up like other specimens, so as to preserve, e. g., the character of "hardpan."

(7) All peculiarities of the soil and subsoil, their behavior in wet and dry seasons, their location, position—every circumstance, in fact, that can throw any light on their agricultural qualities or peculiarities—should be carefully noted and the notes sent with the specimens. Unless accompanied by such notes, specimens can not ordinarily be considered as justifying the amount of labor involved in their examination.

It will be noted that in these "directions" the depth of the surface "soil" sample to be taken is left to the judgment of the farmer, between the limits of 6 and 12 inches, the first being the minimum, the latter the maximum, depth to which rational culture usually reaches.

When the soil sample is to be transmitted to any considerable distance it is very desirable that it should be dried as completely as possible before shipment, since otherwise not only does it often arrive in an abnormally puddled and compacted condition, but may become moldy, changing the natural condition not only of the organic but also of the reducible inorganic ingredients; notably that of ferric hydrate and nitrates. With respect to the latter the date of taking the sample is also desirable and should be recorded in every case.

The Mechanical or Physical Analysis of Soils.

The "insoluble residue" of the chemical analysis indicates in a very general manner the amount of "sand" in a soil; and when combined with a determination of the soluble silica, alumina, and lime we also gain some idea of possible plasticity or "heaviness." But these indications given by chemical analysis are far too indefinite for practical requirements. It is to the mechanical (or physical) analysis that we

must look for practically available data on the tilling qualities of soils; but the loose practice prevailing thus far has rendered the results far from satisfactory.

As regards, first, the conventional "clay" which figures in the analytical statements, *e. g.*, of the German investigators, it oftentimes consists far more of fine silex than even of total kaolinite substance. Since the latter itself consists at least of two portions totally distinct in their physical functions, viz, of chalky crystalloid grains and of a colloidal, highly diffusible, and extremely adhesive and hygroscopic portion, the presence of which determines, in the main, the corresponding physical properties of the soil, the indefiniteness of the current mode of statement and consequent classification of soils is so great as to render a clearer and more rational and definite method of mechanical analysis absolutely necessary from this point of view alone. An additional source of error arises from the viscosity and consequently greater hydraulic efficacy of "clay water," which carries off, at the same velocity, much larger particles of rock powder than does pure water; or, in the case of subsidence, permits them to settle much more slowly. The consequence is that the percentage of a sediment of a given "hydraulic value" will be found very different, according to the amount of colloidal clay present, or according as the latter has or has not been wholly or partially removed prior to the separation of the simply pulverulent or sandy ingredients. Adding to these sources of uncertainty the wide range of size of grain included and weighed as one sediment by most observers, the slight value attaching thus far to the results of mechanical soil analysis as representing the farmer's experience is amply explained.

SEDIMENTATION AND HYDRAULIC ELUTRIATION.

For the separation of fine powders and mixed grain sizes, sedimentation by stirring up in water and drawing off the suspended sediment with the water after a definite time allowed for the subsidence of the coarser portions is, of course, the simplest process, which has been used in the arts and in pharmacy for centuries. For analytical purposes it has the disadvantage that in order to effect a reasonably complete exhaustion of a sediment of a definite size or "hydraulic value" the operation of stirring up and drawing off must be repeated a large number of times, since the time of subsidence is reckoned for the top layer of water while subsidence actually occurs from below as well. The subsidence method requires close and continuous attention in its execution for a length of time proportioned to the minuteness of subdivisions desired; moreover, in the case of fine sediments, which have a tendency to coalesce (flocculate) under the action of irregular currents caused by stirring, the difficulty of obtaining them properly segregated is almost insuperable, greatly detracting from the accuracy of the determinations. The subsidence or (as it has lately been termed) "beaker" method, however convenient on account of its simplicity, is therefore ill adapted to the carrying out of extended investigations requiring the performance of a large number of mechanical soil analyses, and its laboriousness has long caused it to be replaced, in European practice, by various devices embodying, in a more or less perfect form, the idea of separation of grain-sizes by an ascending current of water or "hydraulic elutriation." My personal experience has brought me into full sympathy with this view.

PRELIMINARY EXAMINATION.

The first step toward the determination of the character of a soil sample should always be what might be termed the hand tests, to wit, the observation of the greater or less degree of ease with which the soil lumps crush between the fingers in the dry condition; the presence or absence of coarse sand, etc.; also the change of color on wetting, the degree of rapidity with which the water is absorbed, the extent to which it softens the lumps, and the degree of plasticity assumed when the wet soil is kneaded.

The next step should be the at least approximate identification of the minerals forming the coarse portion, since it must be presumed that, as a rule, these represent the original nature of the fine grains also. We thus already gain a very close insight into the origin and general character of the soil. This mineralogical examination is advantageously combined with an approximate quantitative determination of the coarse portion by sedimentation in a small beaker, or, more advantageously, by means of a "Kuhn's cylinder" and an upward current of water of definite velocity; that of 2 mm. per second is convenient for this purpose, provided that the sample is kept stirred by means of a rod. The dried coarse portion is weighed and then examined with lens and microscope, with or without previous separation by means of Thoulet's or some other dense fluid. This examination is of especial value in the identification of the sample with other soils previously investigated, and thus frequently saves a very large amount of analytical labor.

Detailed mechanical analysis.—When the preliminary examination does not suffice for the purposes in view, and detailed mechanical or "silt analysis" must be resorted to, more elaborate and complex methods and appliances are called for.

When, in 1872, I began the consideration of this subject in connection with the agricultural survey of the State of Mississippi even a very superficial test led me to reject quickly the grossly inaccurate and misleading apparatus of Nöbel, with its four vessels of ever-varying capacity and form and inconsistent head of water. The best then known, that of Schöne, proved much more satisfactory, provided the "colloidal clay" was first removed. But even then there remained a large and variable residual error, shown in the mixed grain sizes of the sediments and correspondingly varying percentage results. A protracted investigation of the causes of these inaccuracies, made in the years 1872 and 1873, and published in the latter year,[1] proved that the great difficulty encountered in the separation of soil ingredients by the ordinary methods of sedimentation heretofore employed has been in the strong tendency of the fine particles to coalesce and settle with the coarser sediments. When violently shaken they part company and become diffused singly through the liquid, which then presents simply a general turbidity, the particles then settling down slowly and singly at the rate corresponding to their individual size or hydraulic value.

The following experiment, well suited to the lecture table, serves to demonstrate the above principle: If a given quantity of pure siliceous sediment of, e. g., 1 mm., hydraulic value (which has therefore been carried off at the velocity of 1 mm. by an ascending current of water) be again placed in the same current, in a long conical tube with cylindrical outlet above, a considerable portion will fail to pass off, and will gather into flocculent aggregates, revolving in the lower (narrow) part of the tube. If instead of the full velocity of 1 mm. only 0.8 mm. is used, none of the sediment will pass off, but after some time will be found wholly gathered together into the heavy flocculent aggregates, when the full velocity of 1 mm. may be used without causing any considerable portion of sediment to be carried off, until by violent stirring with a rod the floccules are destroyed. It is only, however, by repeating this outside stirring a number of times that it is possible to get nearly all the sediment corresponding to the velocity of current actually used to pass off. The stirring by the current itself is powerless to do so, because the return currents down the sides of a conical tube perpetually cause recoalescence or "flocculation."

It is thus clearly established that even with purely siliceous and nonglutinous sediments correct results can not be obtained so long as conical elutriator tubes are employed; and this disposes effectually of the claims of, e. g., Schöne's apparatus (now adopted by the German experiment station) to anything like the accuracy required in this work.

But if cylindrical elutriator tubes are alone admissible then it follows that agitation by outside power, for keeping the soil or powder in suspension and continuously

[1] Amer. Jour. of Sci., Oct. 1873; Proc. Amer. Assn. Adv. Sci., Portland meeting, 1873. See also "The Flocculation of Particles;" Am. J. Sci., March, 1879.

resolving the floccules inevitably formed under the circumstances is indispensible. However, the tendency to coalescence diminishes of course as the size of the grains increases, but does not altogether cease until their diameter exceeds 0.2 mm., or about 16 mm. hydraulic value. For the elutriation of coarser sediments hydraulic stirring may be successfully employed.

We may therefore formulate as follows the conditions to be fulfilled by mechanical soil, analysis upon which definite conclusions as to the physical and agricultural (or "working") qualities of soils and of the functions of the several grain sizes in determining the same may be based:

(1) The preliminary preparation of the soil sample must leave its natural condition unimpaired. It must, therefore, not be heated to any temperature likely to wholly or partially dehydrate any of its constituent colloids; nor must it be triturated or "pestled" in any manner likely to destroy the naturally existing aggregates or floccules cemented by lime carbonate, ferruginous, zeolithic, or other cements. Boiling is free from these objections.

(2) Prior to any attempt to separate the different grain sizes, whether by the hydraulic or subsidence method, all the "colloidal clay" must be completely removed; and in view of the prime importance of the latter as a physical soil ingredient it must be determined by direct weighing, and not merely by loss, or "difference."

(3) If the hydraulic method be used for the separation of the successive grain sizes this must be done in vertical, cylindrical elutriator tubes, provided with a device for mechanical stirring by outside power, for preventing and undoing the flocculation of the sediments into heavy aggregates of indefinite composition and hydraulic value.

(4) The importance of discriminating between the several fine sediments in respect to their physical functions is so great that a much larger number of subdivisions of these must be made than is now usually done. This proviso renders the ordinary process of sedimentation by stirring and subsidence in cylindrical vessels (beaker method) practically inapplicable to any extended investigations requiring frequent and numerous mechanical analyses, since each one would take an amount of time and practice quite out of reach of ordinary laboratory personnel. The work must in the main be done automatically to be generally available.

The ways and means by which these several conditions may most easily be fulfilled will now be considered in detail.

PRELIMINARY PREPARATION.

In some cases simple sifting will serve, without further preparation, to separate the original, dry soil sample into appropriate subdivisions, down to the fine earth that is to serve for detailed mechanical and chemical analysis. In most cases, however, a certain amount of mechanical disintregation must be resorted to in order to detach the earth from the larger sand grains and aggregates, and here some judgment must be exercised by the operator. A preliminary washing, aided by the lens and microscope if necessary, will show whether there are any soft concretions or decomposed rock particles likely to be crushed by a rubber pestle, the hardest material admissible, but which will serve admirably when no soft grains will be crushed by it, thus changing the nature of the soil to a corresponding extent. The liability to such change is much increased by wetting the soil, when calcareous and ferruginous concretions (bog ore) may be crushed to such extent as to destroy the value of the work. In the case of heavy clay ("adobe") soils, however, wetting, and even hot digestion with water, may be necessary to even the preliminary disintegration serving to prepare a fair average sample. The slushy mass must then be economically washed through the fine-earth sieve and the remnant afterwards separated into sizes by sifting, while the fine-earth slush is evaporated and dried to serve for analysis.

Since a sieve with 0.5 mm. mesh is practically about the finest that will serve for the dry separation with advantage, and since that same diameter is almost exactly that of quartz grains passing off at the maximum current velocity conveniently available, viz, 64 mm. per second, I have adopted the 0.5 mm. limit as practically the best for the fine earth to be used for both mechanical and chemical analysis. It is to such fine earth that the data hereinafter given are meant to apply.

DISINTEGRATION BY BOILING.

This is applicable to all soils, the time required varying greatly with different ones. Those containing much lime carbonate require the longest time to resolve those aggregates which will be also destroyed by tillage. Further than this the disintegration should not go; nor should it fall seriously short of that normal measure. Thirty hours have in one case barely sufficed for a black calcareous prairie soil, while three have been found sufficient to reduce other soils to clean, single grains, as observed under the lens. While an absolute rule can not therefore be given it may be said that with most soils from eight to fifteen hours will be the right measure, which with some may extend to twenty and twenty-four. The fact ascertained by Osborne, that the diffusibility of some clays, at least, is diminished by long boiling, renders it desirable to restrict its duration to a minimum.

The boiling is best done in a thin, long-necked flask of about 1 liter capacity, filled four-fifths full of distilled water and laid on a stand, on wire netting, at an angle of 40° to 45°. It is provided with a cork and condensing tube of sufficient length (5 to 6 feet) to condense all or most of the steam formed when ebullition is kept up by means of a gas flame. For a few hours the boiling generally proceeds quietly; but as the disintegration progresses violent bumping sets in, which sometimes endangers the flask, but is of assistance for the attainment of the object in view. In extreme cases some of the heavier sediment (generally clean sand) may be removed from the flask, but this is undesirable.

It is frequently the case that when the boiled contents are left to settle the liquid appears perfectly clear within an hour, although so soon as they are largely diluted the clay becomes diffused as usual, and will not settle in weeks. Probably this is owing to the extraction from the soil of soluble salts, which exert the same influence as does lime or common salt, even in very dilute solutions.

REMOVAL OF THE COLLOIDAL CLAY AND FINEST SEDIMENTS.

The boiled fluid and sediment is transferred to a beaker and diluted so as to form from 1 to 1½ liters in bulk, and, being stirred up, is allowed to settle for such a length of time as (taking into account the height of the column) will allow all sediments of 0.25 mm. hydraulic value to subside, the process being repeated with smaller quantities of water (distilled) until no sensible turbidity remains after allowing due time for subsidence.

It must be remembered that this time is considerably longer than that for pure water, so long as any considerable amount of clay remains in the liquid, rendering it more viscous. And as the precise amount of allowance to be made can not in general be foreseen, some sediment of, and exceeding 0.25 mm. hydraulic value, will almost inevitably be decanted with the successive clay waters, until the buoyant effect of the clay becomes insensible. The united clay waters (of which there will be from 4 to 8 liters) must therefore be again stirred up, and the proper time allowed for the sediments of 0.25 mm. and over to subside. The dilution being very great, a pretty accurate separation is thus accomplished; the sediments being then ready for the elutriator.

SEPARATION OF CLAY AND THE FINEST SILT IN THE " CLAY WATER."

The now well-known property of colloidal clay, of remaining suspended in pure water for weeks and even months, offers an obvious method of separation from at

least the greater portion of silts finer than 0.25 mm. hydraulic value (<0.25). **To** push this separation to the extreme of attempting to remove all but the kaolinite particles proper, even were it feasible, would carry the time and labor required for the determination beyond the limits required for practical purposes, and would render the performance of mechanical analyses rare events in most laboratories. In special cases, of course, it may be desirable to go to these lengths, and also to divide the sediments lying between the clay proper and the finest sediment that can conveniently be obtained by the hydraulic method (0.25 mm. hydraulic value) into two or more groups, when it is very abundant.

I place the clay water for subsidence in a wide cylindrical vessel (in which it may conveniently occupy 200 mm. in height); it is there allowed to settle for twenty-four hours. This interval of time was at first arbitrarily chosen, but I subsequently found it to be about the average time required by the finest siliceous usually present in soils, to sink through 200 mm. of pure water. So long as any sensible amount of clay is present, the time of course is longer, say from forty to sixty hours, or even more, if the clay be abundant and the liquid not very dilute. The sharp line of separation between the dark silt cloud below and the translucent clay water above is readily observed, and the time of subsidence regulated accordingly. At times, several such lines of division may be seen simultaneously in the column, indicating silts of successive sizes, with a break between. No such appearance is presented when, after weeks of quiet, the clay itself gradually settles. The liquid, which may be almost clear at the surface, then shades off downward very gradually, until near the bottom of the vessel, it becomes entirely opaque.

After decantation of the clay water, the remaining liquid is poured off temporarily leaving the sediment as dry as possible. It is then rubbed or kneaded in the decanting vessel itself, with a long-handled, soft-rubber pestle (conveniently cut out of a rubber cork). At this point the addition of a few drops of ammonia water, according to Schölsing's prescription, renders good service; but it is undesirable to use any large amount of ammonia, as it impedes the subsequent precipitation of the colloidal clay.

Distilled water is again poured on (agitating as much as possible to break up the molecular aggregates) to the proper height, and another twenty-four hours' subsidence allowed. This operation is repeated six to nine times, until either the water remains almost clear after the last subsidence, or the decanted turbid water fails to be precipitated by salt water, showing the suspended matter to be pulverulent silt only.

Doubtless the fine silt obtained in the twenty-four hours' subsidence, the diameter of whose quartz particles varies from 0.001 to 0.02 mm. is not entirely free from adherent colloidal clay, as is indicated by its deeper tint, compared with that of the coarser sediments. The extent to which this contamination exists, and the distribution of the important soil ingredients among the several sediments, have been discussed in other papers.

Determination of the colloidal clay.—The colloidal portion of the kaolinite constituent is of such preëminent importance that to throw upon it the indefinite "loss by analysis" and estimate it "by difference," is hardly excusable. Two ways of determining it directly in the turbid waters from the twenty-four hours' subsidences, are open to us. One is to evaporate an aliquot portion; if the latter is not too small, and the soil is measurably free from the carbonates of lime and magnesia and other soluble salts, this method may yield fairly satisfactory results. But 100 cc. out of perhaps 20 liters of water, as has been practiced by some, is at best a rather minute base line to go upon in so important a determination; moreover, it is so desirable to have the "clay" tangibly before one for examination, that I consider it altogether preferable to precipitate it by means of a saline solution and thus weigh the whole. The use of lime water, which naturally suggests itself, is so complicated by chemical reactions and other elements of uncertainty,

that I have found it preferable to employ simply pure, rock-salt brine for the precipitation. Fifty cc. of a saturated brine, i. e., 1.5 per cent of salt, is ordinarily sufficient to precipitate 1 liter of clay water; the precipitation is much favored by warming. Half the quantity, or even less, will do the same, but more time is required, and the precipitate is more voluminous.

In practice it will be found desirable to thus precipitate each lot of clay water as soon as drawn off. The clay precipitate (which greatly resembles the usual iron alumina of chemical analysis) will then at the end of twenty-four hours have shrunk into so small a bulk that on drawing off the supernatant liquid, the succeeding clay water from twenty-four hours' subsidence may be mixed with it, causing it to rediffuse; the same being done at each succeeding drawing-off. This mode of operation greatly facilitates and shortens the gathering of the colloidal clay, which is precipitated much less easily and sharply from very dilute waters than from those heavily charged. As the clay precipitate can not ordinarily be washed with pure water, in which it quickly diffuses, it must be collected on a weighed filter, washed with a weak brine, dried at 100° and weighed. It is then again placed in a funnel and washed with a weak solution of sal ammoniac, until the chlorid of sodium is removed. The filtrate is evaporated, the residue ignited and weighed; its weight, plus that of the filter, deducted from the total weight, gives that of the clay itself.

In some cases, especially of clays and subsoils deeply tinged with iron, the clay, after drying at 100°, will not readily diffuse in water, and can be washed with pure water until free from salt; it can then, of course, be weighed directly.

Properties of pure clay.—The "clay" so obtained is quite a different substance from what usually comes under our observation as such, since its percentage seems rarely to reach 75 in the purest natural clays, 40 to 47 in the heaviest of clay soils, and 10 to 20 in ordinary loams. Thin crusts of it are occasionally found in river bottoms, where clay water has, after an overflow, gradually evaporated in undisturbed pools. When freshly precipitated by salt it is gelatinous, resembling a mixed precipitate of ferric oxid and alumina. On drying, it contracts almost as extravagantly as the latter, crimping up the filter, to which it tenaciously clings, and from which it can be separated only by moistening on the outside, when it may mostly, with care, be peeled off. After drying it constitutes a hard, often horny, mass, difficult to break, and at times somewhat resonant. Since the ferric oxid with which the soil or clay may have been colored is mainly accumulated in this portion, it often possesses a correspondingly dark-brown or chocolate tint. When a large amount of iron is present water acts rather slowly on the dried mass, which gradually swells, like glue, the fragments retaining their shape. Not so when the substance is comparatively free from iron. It then swells up instantly on contact with water; even the horny scales adhering to the upper portion of the filter quickly lose their shape, bulge like a piece of lime in process of slaking, and tumble down into the middle of the filter.

There is a marked difference, however, in the behavior to water of clays equally free from ferric oxid, some exhibiting the phenomena just described in a more energetic manner than others. On the whole, those freest from iron appear to imbibe the water and crumble most readily. As this property possesses highly important bearings, both on the agricultural and ceramic qualities of clays, I propose to investigate it more minutely hereafter.

The pure clay, when dry, adheres to the tongue so tenaciously as to render its separation painful. When moistened and worked into the plastic condition, it is exceedingly tenacious and "sticky," adhering to everything it touches.

Under a magnifying power of 350 diameters no definite particles can be discovered in the opalescent clay water remaining after several weeks' subsidence. The precipitate formed by saline solutions then appears as an indefinite cloud (mostly of a yellowish tinge), for which one vainly seeks a better focus. In stronger clay water, or with higher magnifying powers, one can discern a great number of indefinite

punctiform bodies, very uniformly diffused throughout the liquid, and apparently opaque; the precipitate then formed by brine also shows a faintly dotted structure of its clouds.[1]

Chemical nature of the clay precipitate.—While usually considered as consisting essentially of kaolinite substance in a state of extremely fine division, the colloidal clay doubtless contains in most, if not in all, cases, other colloidals or "hydrogels," whose absorptive functions (albeit not plasticity) are in a measure similar to those of clay. Since in many cases the silica set free by treatment of the precipitate with acid is materially below that of the alumina dissolved by the same treatment, it follows that free aluminic hydrate is then present. The colloidal ferric hydrate, likewise, is accumulated in the clay precipitate, and so are amorphous zeolitic compounds. While it is thus certain that the most careful mechanical separation of this clay can give only an approximation to the really plastic kaolinite substance, yet such approximation is infinitely closer than that attained by determination of total alumina by boiling sulphuric acid, still prescribed in text-books. As in such treatment all the chalky kaolinite particles are also decomposed, it does not lead to even the roughest approximate estimate of the soil's plasticity.[2]

TREATMENT OF THE COARSER SEDIMENTS.

The mixed sediments remaining after the separation of the clay, and of silts of less than 0.25 mm. hydraulic value by decantation, are ready for the elutriator, regarding which some general conditions have already been given; the main point to be guarded being the prevention of the formation of flocculent aggregates out of the granules of the finer sediments.

The elutriator.—The following is a description of the instrument as devised by me for the purpose of breaking up these flocculent aggregates, also of the simpler form (Schöne's elutriator as modified by me), which can serve for grain sizes above 8 mm. hydraulic value (the latter is conveniently selected as to have half the cross section of the former, so that with the same position of the index lever the velocity will be just doubled). A cylindrical glass tube, of about 45 mm. inside diameter at its mouth and 290 to 300 mm. high, has attached to its base a rotary churn, consisting of a brass cup, shaped like an egg with point down, so as to slope rather steeply at base, and triply perforated, viz, at the bottom for connection with the relay reservoir at the sides for the passage of a horizontal axis bearing four grated wings. This axis, of course, passes through stuffing boxes provided with good thick leather washers saturated with mutton tallow. These washers, if the axis runs true, will bear a million or more revolutions without material leakage; when a beginning is noted additional washers may be slipped on, without emptying the instrument, until the analysis is finished. From five to six hundred revolutions per minute is a proper velocity, for the finest sediments, which may be imparted by clockwork, turbine, or electric power. The driving pulley should not be directly connected with the axis, both because it is liable to cause leakage and because it is necessary to be able to handle the elutriator quickly and independently. This is accomplished by the use of "dogs" on the pulley and churn axis. For the grain sizes of 1 to 8 mm. hydraulic value lower velocities are sufficient. Too low a velocity causes an indefinite duration of the operation, and may be recognized by the increase of turbidity as the velocity is increased.

As the whirling agitation caused by the rotation of the dasher would gradually communicate itself to the whole column of water and cause irregularities, a wire screen of 0.8 mm. aperture is cemented to the lower base of the cylinder.

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[1] According to Whitney the finest particles of colloidal clay are 0.0001 mm. diameter; Wollny gives .001 mm. as the limit, stating that the particles have the form of short, straight spicules. Probably the subject is far from its final form as yet.

[2] See, on these points, my article on "the determination of clay in soils." Agr. Science, **6, 156.**

The relay vessel should be a thick, conical test glass with foot. Its object is to serve as a reservoir for the heavy sediments not concerned at the velocity used in the elutriator tube, and whose presence in the latter, or in its base (the churn) would only cause abrasion of the grains and changes of current velocity, such as occur in the apparatus of Schöne, and compel the current measurement of the water delivered. It is connected above with the churn by a brass tube about 10 mm. in clear diameter, so as to facilitate the descent of the superfluous sediments, which the operator, knowing the proportion of area between the connecting tube and elutriator, can carry to any desired extent, thus avoiding the disturbance of the gauged current velocities as well as all material abrasion.

A glass delivery tube should extend quite half way down the sides of the relay vessel to insure a full stirring up of the coarse sediments when required. By means of a rubber hose, not less than 20 inches in length, this delivery tube connects with a siphon carrying the water from near the bottom of a Mariotte's bottle—a 10-gallon acid carboy. A stopcock, provided with a long, stiff index lever moving on an empirically graduated arc, regulates the delivery of water through the siphon. Knowing the area of the cross section of the elutriator tube the number of cubic centimeters of water which should pass through it in one minute at 1 mm. velocity is easily calculated; and from this the lever positions corresponding to other velocities are quickly determined and marked on the graduated arc. The receiving bottle for the sediments must be wide and tall, so as to allow the sediment to settle while the water flows from the top into the waste pipe. The receiving funnel tube must dip nearly to the bottom of the bottle.

Thus arranged the instrument works very satisfactorily, and by its aid soils and clays may readily be separated into sediments of any hydraulic value desired. But in order to insure correct and concordant results it is necessary to observe some precautions, to wit:

(1) The tube of the instrument must be as nearly cylindrical as possible, and must be placed and maintained in a truly vertical position. A very slight variation from the vertical at once causes the formation of return currents, and hence of molecular aggregates on the lower side.

(2) Sunshine, or the proximity of any other source of heat, must be carefully excluded. The currents formed when the instrument is exposed to sunshine will vitiate the results.

(3) The Mariotte's bottle should be frequently cleansed, and the water used be as free from foreign matters as possible. For ordinary purposes it is scarcely necessary to use distilled water. The quantities used are so large as to render it difficult to maintain an adequate supply, and the errors resulting from the use of any water fit for drinking purposes are too slight to be perceptible, so long as no considerable development of the animal and vegetable germs is allowed. Water containing the slimy fibrils of fungoid and moss prothalia, algæ, vorticellæ, etc., will not only cause errors by obstructing the stopcock at low velocities, but these organisms will cause a coalescence of sediments that defies any ordinary churning and completely vitiates the operation.

(4) The amount of sediment discharged at any one time must not exceed that producing a moderate turbidity. Whenever the discharge becomes so copious as to render the moving column opaque the sediments assume a mixed character, coarse grains being apparently upborne by the multitude of light ones, whose hydraulic value lies considerably below the velocity used, while the churner also fails to resolve the molecular aggregates which must be perpetually reforming where contact is so close and frequent. This difficulty is especially apt to occur when too large a quantity of material has been used for analysis, or when one sediment constitutes an unusually large portion of it. Within certain limits the smaller the quantity employed the more concordant are the results. Between 10 and 15 grams is the proper amount for an instrument of the dimensions given above.

THE FINE SEDIMENTS (0.25 TO 4 MM. HYDRAULIC VALUE).

I have found that, practically, 0.25 mm. per second is about the lowest velocity available within reasonable limits of time, and that, by successively doubling the velocities up to 64 mm., a desirable ascending series of sediments is obtained, provided always that a proper previous preparation has been given to the soil or clay. It would seem that, according to the prescription given above for the preliminary sedimentation, no sediment corresponding to 0.25 mm. velocity should remain with the coarser portion. That such is nevertheless always the case, often to a large percentage, emphasizes the difficulty, or rather impossibility, of entirely preventing or dissolving the coalescence of these fine grain sizes by hand stirring, as in "beaker elutriation." It is only by such energetic motion as is above prescribed that this can be fully accomplished, and the delivery of 0.25 and 0.50 mm. hydraulic value really exhausted.

The operation is best begun by turning on a low velocity, 0.25 to 0.50 mm. and then quickly rinsing the sediments from a small beaker into the elutriator before the column reaches the top. The latter is then quickly closed and a few seconds' subsidence allowed with diminishing velocity, so that the turbid column shall not be within less than 30 mm. of the top when the velocity desired is turned on. Otherwise mixed sediments may pass at the beginning. At first the sediment passes off rapidly, and the column remains obviously and evenly turbid from the point where the agitation caused by the churner ceases to the top. But this obvious turbidity generally exhausts itself in the course of a few hours, and it then requires some attention to determine the progress of the operation. I have never known the 0.25 mm. sediment to become properly exhausted in less than fifteen hours, and in one case it has required ninety. The more rigorously the process of preliminary disintegration above described has been carried out the shorter the time required for running off the fine sediments which otherwise tax the operator's patience severely. As a matter of fact they never do give out entirely, doubtless for the reason that the stirrer continues to disintegrate compound particles which had resisted the boiling process. Besides, downward currents on the sides of the vessel will form, despite all precautions, so that the interior surface of the cylinder often becomes coated with pendent flakes of coalesced sediment. These must then from time to time be removed by means of a feather, so as to bring them again under the influence of the stirrer; but it is, of course, almost mathematically impossible that, under these circumstances, any of the sediments subject to coalescence should ever become completely exhausted. Practically, the degree of accuracy attainable at best renders it unnecessary to continue the operation beyond the point when only a milligram or two of sediment comes over with each liter of water. It is admissible and even desirable to run off rapidly the upper third of the column at intervals of fifteen to twenty minutes, whereby not only time is gained, but also the sediment in the reservoir is stirred and brought under the influence of the churner for more complete disintegration.

It is noticeable that recent sediments, river-alluvium, etc., are much more easily worked than more ancient ones, as might be expected. Up to 4 mm hydraulic value the use of the rotary stirrer is indispensable on account of the tendency to the formation of compound particles. Beyond, this tendency measurably disappears, so that for the coarse sediments of 8 to 64 mm. hydraulic stirring may be employed and an elutriating tube of smaller diameter may advantageously be substituted in order to diminish the otherwise somewhat extravagant expenditure of water. The entire amount required for one analysis is from 25 to 30 gallons, provided a thorough previous disintegration has been secured. River water answers in ordinary cases; hard, spring or well waters are undesirable. Distilled water is of course best, and by a simple arrangement of an air-tight reservoir connected with a pressure chamber can be returned to the Mariotte's bottle and used over and over. The average times required for the several sediments are as follows:

Sediment:	Hours.
0. 25 mm	30 to 40
0.5 mm	15 to 25
1.0 mm	5 to 10
2 to 64 mm	6 to 10
Total	56 to 85

With proper arrangements much of this can be done automatically at night, completing an analysis (except the clay and finest silt determinations) in the course of three or four days. Of course only a very small portion of this time is given by the operator to the care of the instrument. He can carry on other work, just as when an evaporation is going on, with only an occasional glance to see that the water supply holds out and that there is no incipient leakage at the axis.

As the soils are most conveniently weighed "dried at 100°,"[1] I have always weighed the sediments in the same condition. Great care is necessary to obtain the correct weight of the extremely hygroscopic clay. The same is true, more or less, of the <0.25 sediment, which, moreover, is so diffusable in water that it can not readily be collected on a filter. I find it best, after letting it subside into as small a compass as possible, to evaporate the last 25–50 cc. in the platinum dish in which it is to be weighed. From the other sediments the water may be decanted so closely as to render their determination easy.

The loss in the analysis of clays and subsoils containing but little organic or other soluble matter is usually from 1.5 to 2 per cent, resulting partially, no doubt, from the loss of the fine silt which comes off, more or less, throughout the process, and is decanted with the voluminous liquid. When the turbidity is marked, it indicates imperfect preliminary disintegration; it may be removed, and the silt collected, by adding a weighed quantity of alum, about 25 milligrams per liter is sufficient, precipitating with ammonia, and deducting from the weight of the flocculent precipitate the calculated amount of alumina.

The analysis of soils very rich in vegetable matter involves some modifications in the preliminary treatment and final weighings, which I shall not discuss here. Ignition of the soil previous to elutriation, as proposed by some, is obviously inadmissible, as it would render impossible the separation of the clay from the finer sediments.

As I have heretofore stated (Am. Jour. Sci., Dec., 1872; Proc. Am. Assoc. Adv. Sci., 1872, p. 71), I consider that, ordinarily, the investigation of the subsoils is better calculated to furnish reliable indications of the agricultural peculiarities of extended regions than that of the surface soils, which are much more liable to local "freaks and accidents," and usually differ from the corresponding subsoils in about the same general points. For practical purposes, therefore, the difficulties incident to the treatment of soils rich in humus may in most cases be avoided.

Character of the sediments.—As regards the size of the particles constituting the successive sediments, the most convenient, because almost universally present, material for reference is quartz sand. I give below a table of measurements, concerning which I remark that the values given refer to the largest and most nearly round quartz grains to be found in each sediment. As a matter of course, all sizes between that given and the one next below are to be found in each sediment. A few grains of the finer sediments are also invariably present, owing both to the progressive disintegration of conglomerated particles by the stirrer and to the inevitable formation of the avalanche-like aggregates of the finer sediments.

While the measurement of the quartz grains (which are rarely wanting in a soil of clay) affords sufficient landmarks to the scientific observer, it seems desirable to

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[1] A somewhat clayey soil will continue to lose weight at 100° for five to six days. But after the first six hours the loss becomes insignificant for the purpose in question.

attach to them, besides, generally intelligible designations, which shall approximately, at least, indicate the nature of the sediment. It is not easy to indicate in popular language distinctions not popularly made, but the grades of grain indicated in the common words grits, sand, and silt, may, if numerically defined, serve at least to establish uniformity of expression among scientific observers and reporters. Thus it might serve a useful purpose to apply the designation of "grits" to all grains above 1 mm. diameter up to "gravel."

Below 1 mm. down to 0.1 mm. might be "sand," all below that "silt," viz: impalpable powders. Then would follow clay, of which the distinctive character is not only impalpable fineness of grain, but also plactisity. To the analyst, however, the designations by hydraulic values will in the nature of the case always remain the most convenient, within the limits of the use of either sedimentation or hydraulic elutriation.

Table of diameters and hydraulic values of sediments.

	Designation of materials.	Diameter of quartz grains.	Velocity per second, or hydraulic value.
		Mm.	*Mm.*
1	Grits............................	1–3	(?)
2	......do..........................	.5–1	(?)
3	Coarse sand......................	.50	64
4	Medium sand	.30	32
5	Fine sand........................	.16	16
6	......do..........................	.12	8
7	Coarse silt	.072	4
8	......do..........................	.047	2
9	Medium silt	.036	1
10	......do	.025	0.5
11	Fine silt	.016	0.25
12	......do..........................	.010	<0.25
13	Clay.............................	....	<0.0023

I remark that the absolute diameter of the elutriator tube exerts a sensible influence on the character of the sediments, in consequence of comparatively greater friction against the sides in a tube of small diameter. Strictly speaking, none of the sediments actually correspond to the velocity calculated from the cross section of the tube and the water delivered in a given time, but to higher ones, whose maximum is in the axis of the tube, and which gradually decrease towards the sides, according to a law which may be demonstrated to the eye by slightly diminishing the velocity while a sediment is being copiously discharged, so that the turbid column remains stationary while clear water is running off. The surface then assumes a paraboloid form, which is sensibly more convex in a tube of smaller diameter than in a wide one; the results obtained in the latter being, of course, nearest the truth.

The sediments are conveniently preserved in homeopathic vials of uniform diameter; these, when arranged in a row, show a surface curve from which the prominent features of the physical composition of a soil may be seen at a glance. The details regarding the interpretation of this curve or of the numerical results with respect to the tilling qualities of soils, and other physical coefficients, I reserve for a future communication.

DETERMINATION OF THE WATER CAPACITY.

This determination as usually made is very indefinite in its results, varying, especially in pervious soils, according to the height of the soil column in which the

water is absorbed. This is due to the obvious fact that at the base of a soil column there is a maximum of this factor, decreasing regularly towards the top of the column, where it becomes a minimum. Now since the total ascent of water in columns of different soils varies from less than 375 mm. to over 1,200 mm., it is clear that any given uniform height of column arbitrarily agreed upon, as proposed by Ad. Mayer and others (e. g., 60 or 200 mm.) will give results standing in no direct rational relation to the maxima and minima of absorption by different soils (see Wollny's Fortschr. Agr. Physik, **15, 1**).

It is evident that in this case, as in others, either a maximum or a minimum determination, or both, should be agreed upon; and such determination should be made in soil columns of as little height as possibe; that is, approaching as nearly as may be to the theoretical postulate of a mere differential. I suggest as the lowest practicable measure for this purpose a column of 10 mm., placed in a circular brass box with perforated bottom, resembling the lead sieve of Plattner's blowpipe chest, and containing exactly 25 cc. In this both the maximum and minimum absorption is determined for each soil, proceeding as follows:

Fill the box full of air-dried soil, of which the moisture content is determined in a separate portion at 100°.[1] Settle the soil by a gentle tapping of the box on a table and then "strike" it level as in measuring grain; weigh.

Place the weighed box, plus soil, on a triangle submerged just beneath the surface of the water in a somewhat wide vessel; allow it to stand until fully saturated (not less than an hour, in order to insure the complete wetting of compacted soil particles); then wipe rapidly with filter paper or an absorbent towel and weigh again without unnecessary delay. Calculate from the weighings the results with respect to both weight and volume.

Now place the same vessel with wet soil on a flat desiccator plate, and immediately cover it with an additional (unweighed) quantity of the same soil, in order to absorb all the water down to the minimum of liquid absorption.

The desiccator plate as well as the bell covering it must be lined with wetted blotting paper in order to maintain a fully saturated atmosphere; and during the latter portion of the process of minimization at least, the soil used must itself have been previously exposed to such an atmosphere long enough to fully saturate with hygroscopic moisture (see above), since otherwise such moisture would be taken up from the wetted soil and would thus vitiate the determination of liquid water capacity. A counterpoised glass plate on the scale pan of the balance serves to cover the soil cylinder while weighing.

After the soil above has become imbued with moisture it is cut off level with the edges of the box by means of a tense, fine silk thread. The upper portion being thrown off, the operation is repeated until the fresh quantities of soil fail to become moistened above the edge of the box and cease to adhere and weight ceases to decrease materially. Lastly, blow off carefully any loose particles remaining on the surface and weigh. Dry the earth, in the box, thoroughly at 100°; weigh. The water thus driven off will represent the minimum of liquid absorption plus the hygroscopic moisture originally contained in the soil, as previously determined by drying at 100°. The latter will, as a rule, be found somewhat greater than that calculated from the first determination, because the unavoidable jarring and the weight of the soil poured on the wet mass compacts the latter to some extent. By simple calculation the determinations may be referred to either of the two quantities for water capacity both by weight and volume.

It is clear that the soil so depleted of the water held in liquid absorption down to the minimum is in precisely the same condition as a similiar layer of soil forming to top of a soil column in which the water has been allowed to rise to its maximum

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[1] The difference arising from drying at 100° as against 200° (see above) is not sufficiently great to influence sensibly the mere approximate results obtainable in the determination of water capacity.

height by capillary ascent. It seems at least probable that hereafter we may be able to deduce from these determinations the absolute height to which water will rise in a soil column in the course of time; but thus far we have no formula for such a deduction. Wollny has shown (Forsch. Agr. Physik, **8**, 197) that, as a general rule, the differences between the maxima and minima become less as the soil becomes more fine-grained; and this difference becomes an important physical datum in judging of the capillary efficiency of the soil. We can then by the above method attain in two or three days results which by direct trial would require at least as many months, and the differences between the maxima and minima thus ascertained far exceed those thus far on record.

METHOD OF CHEMICAL SOIL ANALYSIS.

The methods of chemical soil analysis hereinafter given are essentially those which in their main features have been pursued by Drs. David Dale Owen and Robert Peter in the work of the geological surveys of Kentucky and Arkansas, and which have since been further developed by the writer in the soil work of the surveys of Mississippi and Louisiana; in that done in connection with the Tenth Census "Report on cotton culture" throughout the cotton States; in that of the transcontinental survey, in the States of Oregon, Washington, and Montana, and in the soil work of the California experiment station. Altogether these methods of soil investigation have been applied to over a thousand virgin soils, the analyses of which are strictly comparable among themselves; therefore by far the largest uniform set in existence thus far. Considering the labor involved in the work so performed, it becomes a serious question whether there are any valid reasons why the methods employed in it should be changed in any essential points, since to do so might throw out of any possible comparison with future work the bulk of what has heretofore been done in soil investigation on the North American Continent.

It is for this reason that the methods are given in considerable detail, not with a view of contesting the validity or equal reliability of other processes, but in order that every possible objection may now be raised, before more work shall be placed in jeopardy of being thrown out of comparison by a change of methods. Their convenience and substantial accuracy within the limits of personal error having been tested by considerable experience, and the mode of interpreting the analytical results so as to accord with the farmers' experience in cultivation having been developed with respect to analyses made in accordance therewith, there should be strong reasons for changing the methods before such action is finally taken by this association. In most respects the course of analyses here presented does not differ materially from that laid down by Kedzie in his report on the same subject; the chief differences arise in the preliminary operations, but are there quite vital, so as to necessitate an agreement if the comparability of analyses is to be maintained.

It should be understood that these methods contemplate an approximation to the maximum of solvent effect plants can exert upon the soil, carbonated water being by far too feeble as a solvent to represent even ordinary plant action, while hydrofluoric acid goes far beyond vegetative possibilities.

PRELIMINARY PREPARATION OF THE SAMPLE.

The soil is thoroughly broken up, dry, with a rubber pestle; or in the case of clayey soils is digested with distilled water until fully disintegrated. A weighed quantity (200 to 500 grams) is then sifted or washed, as the case may be, through a sieve of 0.5 mm. clear aperture. If washed, the muddy water must be evaporated to dryness with the soil slush and the whole thoroughly mixed. The sample so obtained constitutes the "fine earth" to be used in chemical analysis. The coarse portions are to be further segregated by sieves, weighed, and their mineralogical constituents identified with the microscope, reagents, or Thoulet's solution, as the case may require.

That the introduction of the grain-sizes coarser than 0.5 mm. can as a rule serve no useful purpose in the chemical analysis, is strikingly shown in the investigation made by R. H. Loughridge, in 1873 (Am. J. Sci., Jan., 1874, p. 17). He found that in the case of a very generalized soil of the Mississippi uplands, solution by strong acid practically ceased beyond the sediment of 0.5 mm. hydraulic value, corresponding to a diameter of about 0.025 mm. Although the general applicability of this particular limit may be fairly questioned, the wide margins between the fractions 0.5 and 0.025 renders it pretty certain that within the limit of the former we shall find all that is of any value to plants for their supply of mineral plant food. But for the sake of comparability with analyses made under a different rule, the grain-sizes of 0.5 to 1 and 1 to 2 mm. diameter should always be quantitatively determined.

DETERMINATION OF THE HYGROSCOPIC COEFFICIENT.

The fine earth is exposed to an atmosphere saturated with moisture for about twelve hours at the ordinary temperature (60° F.) of the cellar in which the box should be kept. For this it is sifted in a layer of about 1 mm. thickness upon glazed paper, on a wooden table in a small water-tight covered box (12 by 9 by 8 inches) in which there is about an inch of water; the interior sides and cover of the box should be lined with blotting paper, kept saturated with water, to insure the saturation of the air.

"Air-dried soil" yields results varying from day to day to the extent of as much as 30 to 50 per cent, nor have we any corrective formula that would reduce such observations to absolute measure. Knop's law, that the absorption varies directly as the temperature, while applicable to low percentages of saturation, is wide of the truth when saturation is approached. My observation has shown that between the temperature of about 7 and 23° C. the coefficient of absorption in saturated air varies only by a small fraction; hence the ordinary temperature of cellars will serve well in these determinations without material correction.

After eight to twelve hours the earth is transferred as quickly as possible, in the cellar, to a weighed drying tube (see Fig. 1) and weighed. The tube is then placed in a paraffin bath; the temperature gradually raised to

FIG. 1.

200° C. and kept there twenty to thirty minutes (rapidity of raising temperature depending upon the amount of moisture in the soil), a current of dry air passing continually through the tube. It is then weighed again, and the loss in weight gives the hygroscopic moisture in saturated air.

The reason for adopting 200° C. as the temperature for drying instead of 100° is that water will continue to come off from most soil at the latter temperature for an indefinite time—a week or more—before an approach to constancy of weight is attained; and that up to 200° only an arbitrary limit can be assigned for the expulsion of hygroscopic moisture. Moreover, the great majority of soils (those poor in humus) will reabsorb moisture from a saturated atmosphere to the full extent of that driven off at 200° C. These observations, originally made by Dr. David Dale Owen, I have since repeatedly verified.

GENERAL ANALYSIS.

The samples for general analysis and phosphoric acid determination are weighed out directly from the drying tube.

In determining the amount of material to be employed for the general analysis regard must be had to the nature of the soil. This is necessary because of the impracticability of handling successfully such large precipitates of alumina as would result from the employment of as much as 5 grams in the case of calcareous clay soils; while in the case of very sandy soils even that quantity might require to be doubled in order to obtain weighable amounts of certain ingredients. For average loam soils in which the soluble portion ranges from 60 to 80 per cent, 2.5 to 3 grams is about the right measure for general analysis, while for the phosphoric acid deter-

mination not less than 3 grams should be employed in any case. It has been alleged that larger quantities must be taken for analysis in order to secure average results. It is difficult to see why this should be true for soils and not for ores, in which the results affect directly the money value, while in the case of soils the interpretation of results allows much wider limits in the percentages. Correct sampling must be presupposed to make any analysis useful; but with modern balances and methods it is difficult to see why 5 grams should be employed instead of half that amount, which in some cases is still too much for convenient manipulation of certain precipitates.

(1) The weighed quantity, usually of 2 to 2.5 grams, is brought into a small porcelain beaker covered with a watch glass, treated with 8 to 10 times its bulk of hydrochloric acid of 1.115 sp. gr. and 2 or 3 drops of nitric acid, and digested for five days over the laboratory steam bath. At the end of this time it is evaporated to dryness, first on the water bath and then on the sand bath. By this treatment all the silica set free is rendered insoluble.

In an investigation made by Loughridge in 1873 (Am. J. Sci. Jan., 1874, p. 20) it was found that acid of the above strength exerted a higher solvent power than that materially stronger or weaker (1.100 or 1.160 sp. gr.). The above standard strength is easily prepared by steam distillation of acid either stronger or weaker. He also found that after the fifth day no further essential solvent action occurred. Later investigations by Jaffa show that the effect of such acid approximates closely to that of oxalic acid, the strongest solvent available for plant-root action. We therefore determine in such treatment the maximum effect vegetation can exert on soils. It is now moistened with strong hydrochloric acid and 2 or 3 drops of nitric acid, warmed, and, after allowing it to stand a few hours on the water bath, treated with distilled water. After clearing it is filtered from the insoluble residue, which is strongly ignited and weighed. If the filtrate should be turbid the insoluble residue which has gone through the filter can be recovered in the iron and alumina determination.

The insoluble residue is next boiled for fifteen or twenty minutes in a concentrated solution of carbonate of soda, to which a few drops of caustic lye should then be added, to prevent reprecipitation of the dissolved silica. The solution must be filtered hot. The difference between the weight of the total residue and that of undissolved sand and mineral powder is recorded as soluble silica, being the aggregate of that set free by the acid treatment and that previously existing in the soil. The latter, however, rarely reaches, 5 per cent.

(2) The acid filtrate from the total insoluble residue is evaporated to a convenient bulk. In case the filtrate should indicate by its color the presence of any organic matter, it should be oxidized by aqua regia, otherwise there will be difficulty in separating alumina.

(3) The filtrate thus prepared is now brought to boiling and treated sparingly with ammonia, whereby iron and alumina are precipitated. It is kept boiling until the excess of ammonia is driven off, and then filtered hot. (Filtrate A.) The previous addition of ammonic chlorid is usually unnecessary. If the boiling is continued too long filtration becomes very difficult and a part of the precipitate may redissolve in washing. Filtration may be begun so soon as the nose fails to note the presence of free ammonia; test paper is too delicate. Failure to boil long enough involves the contamination of the iron-alumina precipitate with lime and manganese.

(4) The iron and alumina precipitate (with filter) of No. 3 is dissolved in a mixture of about 5 cc. hydrochloric acid and 20 cc. water. Then filter and make up to 150 cc. Take 50 cc. for the determination of iron and alumina together by precipitation with ammonia, after oxidizing the organic matter (filter) with aqua regia; also 50 cc. for iron alone; keep 50 cc. in reserve. Determine the iron by means of a standard solution of permanganate of potash after reduction; this latter is done by evaporating the 50 cc. almost to dryness with strong sulphuric acid, adding water and trans-

ferring the solution to a flask, and then reducing by means of pure metallic zinc in the usual way. The alumina is then determined by difference. This method of determining the two oxids in their intermixture is in several respects more satisfactory than the separation with alkaline lye, which, however, has served for most determinations made until within the last ten years. It is much more liable to miscarry in unpracticed hands than the other.

(5) The filtrate A from iron and alumina is acidified slightly with HCl, and if too bulky is evaporated down to about 25 cc. (unless the soil is a very calcareous one) and the lime is precipitated from it by neutralizing with ammonia and adding ammonic oxalate. The precipitation of the lime should be done in the hot solution, as the precipitate settles much more easily. It is allowed to stand for twelve hours, then filtered off, washed with cold water, and dried (filtrate C). By ignition the lime precipitate is partially converted into the oxid. It is then heated with excess of powdered ammonium carbonate, moistened with water, and exposed to a gentle heat (50°–80° C.) until all the ammonia is expelled. It is then dried below red heat and weighed as lime carbonate. When the amount of lime is at all considerable, the treatment with ammonic carbonate must be repeated till a constant weight is obtained.

(6) The filtrate B from the calcic oxalate is put into a hard Bohemian flask, boiled down over the sand bath and the ammoniacal salts destroyed with aqua regia (Lawrence Smith's method). From the flask it is removed to a small beaker and evaporated to dryness with excess of HNO_3. This process usually occupies four to five hours. The residue should be crystalline-granular; if white-opaque, ammonic nitrate remains and must be destroyed by HCl.

The dry residue is now moistened with nitric acid and the floccules of silica usually present separated by filtration from the filtrate, which should not amount to more than 10 or 15 cc. SO_3 is then precipitated by treatment with a few drops of baric nitrate, both the solution and the reagent being heated to boiling. If the quantity of sulphuric acid is large, it may be filtered off after the lapse of four or five hours (filtrate C). If very small let it stand twelve hours. The precipitate is washed out with boiling water, dried, ignited, and weighed (filtrate C). Care should be taken in adding the barium nitrate to use only the least possible excess, because in such a small concentrated acid solution the excess of barium nitrate may crystallize and will not readily dissolve in hot water. Care must also be taken not to leave in the beaker the large heavy crystals of baric sulphate, of which a few sometimes constitute the entire precipitate, rarely exceeding a few milligrams. Should the ignited precipitate show an alkaline reaction on moistening with water, it must be treated with a drop of HCl, refiltered and weighed. The use of barium acetate involves unnecessary trouble in this determination.

(7) Filtrate C is now evaporated to dryness in a platinum dish; the residue is treated with an excess of crystallized oxalic acid, moistened with water, and exposed to gentle heat. It is then strongly ignited to change the oxalates to carbonates. This treatment with oxalic acid must be made in a vessel which can be kept well covered, otherwise there is danger of loss through spattering. As little water as possible should be used, as otherwise loss from evolution of carbonic gas is difficult to avoid. Spatters on the cover should not be washed back into the basin until after the excess of oxalic acid has been volatilized. The ignited mass should have a slightly blackish tinge to prove the conversion of the nitrates into carbonates. White portions may be locally retreated with oxalic acid. The ignited mass is treated with a small amount of water, which dissolves the alkaline carbonates and leaves the carbonates of magnesia, protosesquioxid of manganese, and the excess of barium carbonate behind. The alkalies are separated by filtration into a small platinum dish (filtrate D), and the residue is well but economically washed with water on a small filter. When the filtrate exceeds 10 cc., it may on evaporation show so much turbidity from dissolved earthy carbonates as to render refiltration on a

minute filter necessary, since otherwise the soda percentage will be found too large, magnesia too small. If on dissolving the ignited mass the solution should appear greenish from the formation of alkaline manganates, add a few drops of alcohol to reduce the manganese to insoluble dioxid. The residue of barium, magnesium, and manganese compounds is treated on the filter with hydrochloric acid, and the platinum dish is washed with warm nitric acid (not hydrochloric, for the platinum dish may be attacked by chlorin from the manganese oxid) dissolving any small traces of precipitate that may be left behind.

(8) The solution containing the chlorids of magnesium and manganese is freed from the barium salts by hot precipitation with sulphuric acid, and the barium sulphate after settling a few hours is filtered off. The filtrate is neutralized with ammonia, any resulting small precipitate (of iron) is filtered off, and the manganese precipitated with ammonic sulphid. Let stand twelve hours and filter (filtrate E); wash with cold water, dry, ignite, and weigh as manganese protosesquioxid Mn_3O_4. If preferred the manganese may be precipitated with chlorin or bromin water as dioxid; but the process requires a rather longer time and may fail in inexpert hands more readily than the other.

(9) The filtrate E from the manganese is now freed from sulphur by acidulating with hydrochloric acid, evaporating down, if necessary, and filtering. From the filtrate the magnesia is precipitated by adding an equal bulk of ammonia water and then sodic phosphate. After standing at least twenty-four hours, the magnesia salt may be filtered off, washed out with ammoniacal water, dried, ignited, and weighed as magnesium pyrophosphate.

(10) The filtrate D, which should not be more than 10 or 15 cc., containing the carbonates of the alkalies, is evaporated to dryness and gently fused, so as to render insoluble any magnesium carbonate that may have gone through; then redissolved and filtered into a small weighed platinum dish containing a few drops of dilute hydrochloric acid, to change the carbonates into chlorids; evaporated to dryness, exposed to a gradually rising temperature (below red heat), by which the chlorids are thoroughly dried and freed from moisture, so as to prevent the decrepitation that would otherwise occur on ignition. Then, holding the platinum basin firmly by forceps grasping the clean edge, pass it carefully over a very low Bunsen flame, so as to cause, successively, every portion of the scaly or powdery residue to collapse, without fully fusing. There is thus no loss from volatilization, and no difficulty in obtaining an accurate, constant weight. The weighed chlorids are washed by means of a little water into a small beaker or porcelain dish, treated with a sufficient quantity of platinic chlorid, and evaporated to dryness over the water bath. The dried residue is treated with a mixture of 3 parts absolute alcohol and 1 part ether, leaving the potassio-platinic chlorid undissolved. This is put on a filter, and washed with ether-alcohol. When dried, the precipitate and filter are put into a small platinum crucible and exposed to a heat sufficiently intense to reduce the platinum chlorid to metallic platinum and to volatilize the greater part of the potassium chlorid. This is easily accomplished in a small crucible, which is roughened by being constantly used for the same purpose (and no other), the spongy metal causing a ready evolution of the gases. (See Fres. Ztschr. anal. Chem., 1893.) The reduced platinum is now first washed in the crucible with hot acidulated water, then with pure water; then all moisture is driven off and it is weighed. From the weight of the platinum is calculated the potassic chlorid and the oxid corresponding; the difference between the weights of the total alkalin chlorids and potassic chlorid gives the sodic chlorid, from which may be calculated the sodic oxid. When the heating of the platinum precipitate has not been sufficient in time or intensity, instead of being in a solid spongy mass of the color of the crucible itself, small black particles of metallic platinum will obstinately float on the surface of the water in the crucible, and it becomes difficult to wash without loss.

Phosphoric Acid Determination.

(11) The weighed quantity (usually of 3 to 5 grams) is ignited in a platinum crucible, care being taken to avoid all loss by dusting. The loss of weight after full ignition gives the amount of chemically-combined water and volatile and combustible matter.

(12) The ignited soil is now removed to a porcelain or glass beaker treated with four to five times its bulk of strong nitric acid, digested for two days, evaporated to dryness first over the water bath and then over the sand bath, moistened with nitric acid, heated and treated with water. After standing a few hours on the water bath it is filtered off from the insoluble residue and the filtrate is evaporated to a very small bulk (10 cc.), and treated with about twice its bulk of the usual ammonium molybdate solution, thus precipitating the phosphoric acid. After standing at least twelve hours, at first at a temperature of about 50° C., it is filtered off and washed with a solution of ammonium nitrate acidified with nitric acid. The washed precipitate is dissolved on the filter with dilute ammonia water. After washing the filter carefully the ammoniacal solution is treated with magnesia mixture, by which the phosphoric acid is precipitated. After allowing it to stand twenty-four hours it is filtered off, washed in the usual way, dried, ignited, and weighed as magnesium pyrophosphate, from which the phosphoric acid is calculated. The per cent of phosphoric acid found is to be subtracted from that of the alumina. When a gelatinous residue remains on the filter after dissolving the molybdo-phosphate with ammonia it may consist either of silica not rendered fully insoluble in the first evaporation, or, more rarely, of alumina containing phosphate. It should be treated with strong nitric acid, and the filtrate with ammonic molybdate; any precipitate formed is of course added to the main quantity before precipitating with magnesia solution.

Humus Determination in Soils.

(*Grandeau's method.*)

About 10 grams of soil is weighed off into a prepared filter. The soil should be covered with a piece of paper (a filter) so as to prevent it from packing when solvents are poured on it. It is now treated with hydrochloric acid from 0.5 per cent to 1 per cent strong (25½ cc. of strong acid and 808 cc. of water) to dissolve out the lime and magnesia which prevent the humus from dissolving in the ammonia. Treat with the acid until there is no reaction for lime; then wash out the acid with water to neutral reaction. Dissolve the humus with weak ammonia water, prepared by diluting common saturated ammonia water (178 cc. ammonia to 422 cc. water). Evaporate the humus solution to dryness in a weighed platinum dish at 100° C.; weigh, then ignite; the loss of weight gives the weight of humus.

The residue from ignition is carbonated with carbonic gas, heated and weighed, thus giving the ash. It is then moistened with nitric acid and evaporated to dryness. The residue is treated with nitric acid and water, allowed to stand a few hours, and the solution filtered from the insoluble residue, which is ignited and weighed, giving the silica.

The soluble phosphoric acid is determined from the solution by the usual method, as magnesic pyrophosphate. It usually amounts to a fraction varying from one-half to as little as one-tenth of the total in the soil. While the P_2O_5 so determined is manifestly more soluble and more available to vegetation than the rest of that found by extraction with stronger acid, it is clearly not as available as that which, when introduced in the form of superphosphates, exerts such striking effects even though forming a much smaller percentage of the whole soil. Nevertheless, very striking agreement with actual practice is often found in making this determination.

The estimation of humus by combustion, in any form, of the total organic matter

in the soil gives results varying according to the season and having no direct relation to the active humus of the soil. The same objection lies against extraction with strong caustic lye.

DETERMINATION OF NITROGEN IN SOILS.

The humus determination will in the case of virgin soils usually indicate approximately the store of nitrogen in the soil, which must be gradually made available by nitrification. Ordinarily (outside of the arid regions) the determination of ammonia and nitrates present in the soil is of little interest for general purposes, since these factors will vary with the season and from day to day. Kedzie (in the report above quoted) proposes to estimate the active soil nitrogen (ammonia plus nitrates and nitrites) by treatment of the whole soil with sodium amalgam and distillation with lime. The objection to this process is that the formation of ammonia by the reaction of the alkali and lime upon the humus amides would greatly exaggerate the active nitrogen and lead to a serious overestimate of the soil's immediate resources.

The usual estimate of nitrogen in black soil-humus (Grandeau's *matiere noire*, determined as above) is from 6 to 8 per cent in the regions of summer rains. From late determinations it would seem that in the arid regions the usually small amount of humus (often less than 0.20 per cent) is materially compensated by a higher nitrogen percentage. It thus becomes necessary to determine the humus-nitrogen directly; and this is easily done by substituting in the Grandeau process of humus-extraction potash or soda lye for ammonia water, and determining the nitrogen by the Kjeldahl method in the filtrate. While it is possible that the ammonia water would not vitiate the determination of nitrogen in the case of neutral or calcareous soils, it would certainly do so in the case of those having an acid reaction.

The lye used should have the strength of 4 per cent in the case of potassic hydrate, 3 per cent in that of sodic hydrate. The black humus filtrate is carefully neutralized with sulphuric acid, evaporated to a small bulk in a beaker or evaporating basin, and the reduced liquid finally evaporated to dryness in the Kjeldahl flask itself by means of a current of air. The beaker or basin is washed either with some of the alkaline lye, or, after evaporation, with warm concentrated sulphuric acid, which is then used in the nitrogen determination in the usual way.

For the determination of nitrates in the soil it is, of course, usually necessary to use large amounts of material, say not less than 100 grams and, according to circumstances, five or more times that amount. In the evaporated solution the nitric acid is best determined by the reduction method, as ammonia.

Usually the soil-filtrate is clear and contains no appreciable amount of organic matter that would interfere with the determination; yet in the case of alkaline soils (impregnated with carbonate of soda) a very dark-colored solution may be obtained. In that case the soil may advantageously be mixed with a few per cent of powdered gypsum before leaching; or the gypsum may be used in the filtrate to discolor it by the decomposition of sodic carbonate and the precipitation of calcic humate. The evaporated filtrate can then be used for the nitrate determination by either the Kjeldahl, Griess, or the Nesslerizing process, which will, of course, include such portions of the ammoniacal salts as may have been leached out.

For the separate determination of these and of the occluded ammonia, when desired, it is probably best to mix the wetted soil intimately with about 10 per cent of magnesic oxid and distill off into titrated chlorhydric acid. For general purposes, however, this determination is usually of little interest.

It is strenuously suggested that in the presentation of the results of a soil analysis the order of the electrolytic series be observed, as in the schedule annexed, so as to facilitate comparisons. The insoluble residue is best placed at the head of the column, as it indicates at a glance, approximately, the general character of the soil as sandy or clayey.

For suggestions concerning the interpretation of analyses made according to the above methods, see "Report on the Experiment Stations of the University of California," 1890, pages 151 to 172.

Coarse materials $> 0.5^{mm}$ ....

Fine earth ....

ANALYSIS OF FINE EARTH.

Insoluble matter .... }

Soluble silica .... }

Potash (K_2O) ....

Soda (Na_2O) ....

Lime (CaO) ....

Magnesia (MgO) ....

Br. ox. of manganese (Mn_3O_4) ....

Peroxide of iron (Fe_2O_3) ....

Alumina (Al_2O_3) .... ·

Phosphoric acid (P_2O_5) ....

Sulphuric acid (SO_3) ....

Carbonic acid (CO_2) ....

Water and organic matter ....

   Total ....

Humus ....

Ash ....

  Sol. phos. acid ....

  Silica ....

Nitrogen (organic) ....

Hygroscopic moisture ....

 Absorbed at .... $°C$

                Vol.   W't.

Water capacity:

  Max ....

  Min ....

Mr. Kedzie read the following paper:

VESSELS FOR ACID-DIGESTION OF SOILS.

By R. C. ...

The kind of vessel in which to carry on acid digestion of soils preparatory to their chemical analysis having called out some discussion, permit me to say a few words on the subject.

If the chemist could find vessels absolutely "unattackable" by acids, the problem would be solved. In the absence of substance of this class, the next best vessel would be one of a material but slightly affected by simple acids and yet foreign to the constituents of ordinary soils. Platinum fills this place best of all, but the expense of platinum beakers for acid digestion of soils would forbid their use in most laboratories.

For cheaper materials for this use we turn to vessels of Berlin porcelain and Bohemian glass, because they better withstand the action of acids than other materials of their class. Yet, even vessels of this class are not "unattackable," since even distilled water will dissolve a small amount. Prof. Johnson, in his appendix to Johnson's Fresenius, found that distilled water boiled in a flask of Bohemian glass and the evaporation, completed in a platinum dish, left on the average a residue of 14 milligrams to the liter, while Berlin porcelain left only one-half of a milligram to the liter of water.

In the second experiment with distilled water, containing 10 per cent HCl boiled down in Bohemian flask, and the drying completed in a platinum dish, no residue was left beyond that left by the original water, and "dilute HCl had not attacked the glass." Dilute acid, treated in the same way with vessel of Berlin porcelain, left .0053 grams of solids per liter in excess of that contained in distilled water.

Fresenius Quantitative Analysis, 16th ed., p. 65, says: "Dilute acids, though exercising no very powerful action on porcelain, yet attack that material more strongly than glass."

Chemists are aware that acids stored for a long time in crown-glass bottles dissolve enough of the material to require a correction in very exact work. The "C. P. HCl sp. gr. 1.115," used for soil analysis stored for a month in crown-glass bottle, on evaporation in a platinum dish for each 50 cc. of acid, left a residue of .00535 gr. and a correction for this residue was made in the following trials.

Three separate trials, each in duplicate, were made to determine the amount of matter dissolved from Bohemian glass bottles and from beakers of Berlin porcelain, when heated with 50 cc. of HCl, sp. gr. 1.115 to temperature of the steam bath for 36 hours, and then evaporating the acid residue to dryness in platinum dish.

The corrected results were as follows:

Residue.	Bohemian glass.	Berlin porcelain.
	Grams.	*Grams.*
First trial	.0030	.0070
Second trial	.0028	.0058
Third trial....................................	.0023	.0041
Average....................................	.0027	.0056

From these results it appears that this acid dissolves a little more than twice as much from Berlin porcelain as from Bohemian glass.

The residues from the second trial were found to contain .00136 grams SiO_2 from Bohemian glass and .00146 from Berlin porcelain. They contained enough Na and Ca to give distinct spectra by flame reaction—the Na very abundant; also a trace of iron. After precipitating SiO_2, the aqueous filtrate was evaporated with $PtCl_4$ and .0007 grams of the double chlorid of potassium, and platinum were obtained from the glass residue, and .0014 grams from porcelain residue, or twice as much potash from porcelain as from glass. The amount, however, was small, and the error from this source in the analysis of five grams of soil would not exceed the fourth place of decimals in a percentage estimate of results.

When it is remembered that the material for making porcelain almost always contain potash, and that the glazing often consists of a mixture of feldspar and quartz, and must contain some alkaline materials to make a mixture more fusible than the biscuit—a fusible glass in fact—we need not wonder at finding a small amount of potash in the acid residue of digestion in porcelain beakers.

The cost of this copper bath for digestion was $1, so that the expense is not an objection to its use. When once mounted and digestion is begun no attention is required beyond the supply of water in the bath, a matter easily secured by inverting a bottle of water with a tube leading nearly to the bottom of the bath.

Mr. Huston read the following paper:

A MODIFICATION OF GRANDEAU'S METHOD FOR THE DETERMINATION OF HUMUS.

By H. A. Huston and F. W. McBride.

Early in the history of soil analysis the importance of the determination of the amount of organic matter in the soil attracted attention. The earlier methods were based upon the determination of the loss on ignition or on the determination of the total carbon by oxidation methods—combustion with copper oxid, oxidation with chromic acid, or with alkaline permanganates. The carbonaceous matter was calculated from the total carbon present on the assumption that the organic matter of the soil contained 58 per cent of carbon, although percentages varying from 40 to 72 had been found by different workers.

It was soon recognized that all the organic matter in the soil was not in the same condition, but existed in various stages of oxidation. There is a general opinion that the black or dark brown material of the soil, resulting from the decay of vegetable matter, has a much higher agricultural value than undecomposed vegetable matter. No very sharp dividing line can be drawn, for changes in the soil are continually going on, and material may be found in almost every stage between cellulose and carbon dioxid. The character of the intermediate products will vary according to the conditions of temperature and tillage and the supply of air and water.

For agricultural purposes some means of determining the amount of decomposed matter (humus) is very desirable. Several solvents have been tried for this purpose. The earlier attempts were made by treating the soil with successive quantities of boiling half saturated solution of carbonate of soda until the soil appeared to yield no more coloring matter to the solvent. The solutions were then united, rendered acid with HCl, which precipitated the humic acid, which was then washed, dried, and weighed. This was considered the more soluble portion of the humic acid. The soil was afterward treated with boiling caustic potash solution in the same manner and the humus extracted was considered insoluble humus. This last process was really more in the nature of manufacturing humus; for sawdust treated with caustic potash solution yields humus, and the inert organic matter of the soil was decomposed to some extent by the caustic alkali; neither of the processes provided for the separation of the humic acid from the lime, magnesia, alumina, and iron, with which it is usually combined in the soil.

The process devised by Grandeau provides for this separation and is the one meeting with the most favor at present. As furnished us by Dr. Hilgard, in April, 1892, it is as follows:

"The moisture content of the sample must be determined in a separate portion. Ten grams are placed in a funnel provided with a filter, the surface is covered with filter paper, and earth washed with HCl of 2 per cent, until the filtrate gives no more reaction of Ca. with ammonic oxalate. Then wash with distilled water until acid reaction ceases fully. Then follow with dilute ammonia water, say 4 per cent NH_3. This will go through very dark colored at first, gradually decreasing; wash until filtrate is nearly or quite colorless. Keep evaporating the filtrate *pari passu* with the washing, in a weighed platinum basin. After all is evaporated, weigh, "dried at 100°". Then ignite; the loss by ignition represents the "humus," distinct from all the unhumified vegetable débris, which would otherwise often increase the apparent humus contents to double of what it really is; but which is always included in the results either of combustion or by extraction with caustic lye. The ash remaining after combustion contains the soluble phosphates (potash, lime, and magnesia) that were associated with the humus. I usually determine the P_2O_5 at least. Care must be taken not to move the soil during the washing-out process, as it can easily be puddled so as to render complete extraction impossible.".

The process recommended by Dr. Hilgard in November, 1892, is as follows:

"About 10 grams of the soil are weighed off into a prepared filter. The soil should be covered with a piece of paper (filter), so as to prevent it from packing when solvents are poured on it. It is now treated with hydrochloric acid from 0.5 per cent to 1 per cent strong ($25\frac{1}{2}$ cc. of strong acid and 808 cc. of water), to dissolve out the lime and magnesia which prevent the humus from dissolving in ammonia. Treat with the acid until there is no reaction for lime; then wash out the acid with water to neutral reaction. Dissolve the humus with weak ammonia water prepared by diluting common saturated ammonia water (178 cc. ammonia to 422 cc. water). Evaporate the humus solution to dryness in a weighed platinum dish at 100° C.; weigh, then ignite; the loss of weight gives the weight of humus. The residue from ignition is carbonated with carbonic acid gas, heated and weighed; thus giving the ash. It is then moistened with nitric acid and evaporated to dryness. The residue is treated with nitric acid and water, allowed to stand for a few hours, and the solution filtered from the insoluble residue, which is ignited and weighed, giving the silica.

The "soluble" phosphoric acid is determined from the solution by the usual method, as magnesia pyrophosphate. It usually amounts to a fraction, varying from one-half to as little as one-tenth of the total in the soil. While the P_2O_5 so determined is manifestly more soluble and more available to vegetation than the rest of that found by extraction with stronger acid, it is clearly not as available as that which, when introduced in the form of superphosphates, exerts such striking effects even though forming a much smaller percentage of the whole soil. Nevertheless, a very striking agreement with actual practice is often found in making this determination. The estimation of "humus" by combustion, in any form, of the total organic matter in the soil, gives results varying according to the season, and having no direct relation to the active humus of the soil." The theory of the process is that the preliminary treatment of the soil with HCl releases the humic acid from its compounds and leaves it free to combine with the ammonia to form the readily soluble humate of ammonia.

It will be noticed that in the latter set of directions the strength of the acid has been reduced to less than one-half and the strength of ammonia solution has been nearly doubled, as the solution last given contains approximately 7.3 per cent of ammonia. No reason is given for this change in the strength of the solutions used. On trying the method it was noticed that with some of the black soils of Indiana the ammonia continued to extract coloring matter for a very long time, and the extraction required in some cases over a week. Duplicate samples of the same soil permitted the ammonia to flow through them at different rates, and there was always an uncertainty regarding the end of the operation and next to an impossibility of exactly repeating the work under similar conditions.

In case results of different workers are to be compared, it is of the greatest importance that methods should be used that are of such a nature that errors resulting from difference of manipulation and from difficulty of reproducing duplicate work can be reduced to a minimum. Hence a simple modification of the Grandeau method was tried, which has the advantage of keeping a definite amount of the soil in contact with a definite volume of ammonia for a fixed time, the strength of the ammonia remaining constant, and the air excluded. The soil is washed with acid and water, as usual; the soil is then washed into a 500 cc. glass-stoppered cylinder, with 500 cc. ammonia, the cylinder closed, and well shaken, and allowed to remain for a definite time, usually thirty-six hours. The material is shaken at regular intervals. The cylinder is left inclined as much as possible without having the fluid touch the glass stopper, thus allowing the soil to settle on the side of the cylinder and exposing a very large surface to the action of the ammonia. During the last twelve hours the cylinder is placed in a vertical position, to allow the soil to settle well before taking out an aliquot part of the solution.

The following points were under investigation:

(1) Comparison of the Grandeau method with this modified method.

(2) Influence of varying the strength of the ammonia used.

(3) Influence of varying the time of digestion.

(4) Influence of varying the temperature of digestion.

(5) Is it possible to complete the extraction by Grandeau's method in a reasonable time?

(6) Comparison of differences of duplicates by each method.

(7) Are the amounts of phosphoric acid, potash, etc., found in the ash necessarily associated with the humus, as claimed, or are they to be ascribed to the solvent action of the ammonia and to changes due to the absorptive properties of soils?

The process of washing the soil with acid, water, and ammonia is very tedious when performed in the usual way with the wash bottle. A simple automatic washing apparatus was devised by which a fixed volume of the washing fluid can be delivered at regular intervals, giving time for thorough draining between each addition of the fluid, and requiring very little attention. The essentials of the apparatus are a large bottle to hold the washing solution, a siphon to the lower end of which is attached a stopcock; to the lower end of this stopcock is attached, by means of a cork with slot cut in the sides, a glass tube 13 cm. long 13 mm. in diameter. The lower end of this tube is closed by a rubber stopper, through which passes a glass tube of 2 mm. bore, which is turned back upon itself inside the larger tube, forming an intermittent siphon, which delivers about 6 cc. of fluid. The funnels containing the soils are placed immediately below the lower end of the siphon. By means of T tubes the washing fluid may be delivered to as many samples as may be desired. The stopcocks are so adjusted that all the fluid may drain through the soil before the siphon fills and delivers another amount of the washing fluid. Instead of washing on the usual form of filter paper in funnels, I prefer with this apparatus to hold the soils on disk of filter paper resting on perforated porcelain plates placed in the funnels. The soil is covered with a disk of filter paper, and small pieces of glass rod are used to hold down this filter paper. This form of apparatus removes the necessity of washing out large filter papers, does not permit the accumulation of humus on the edges of the filter when the Grandeau process is used, and insures that all the washing fluid collected in the flask below shall pass through the soil and not around it. In all cases the acid passed readily through the soil. The water following the acid passed through the soil fairly well until the soil was practically free from chlorids; but in case of certain soils containing fine clay the water passed very slowly after the soil was nearly free from chlorids. With these soils, and also with some muck, the ammonia would not pass through the soil after two or three quantities had been delivered by the siphon. As no trouble had been experienced with the filters folded in the usual form, an examination was made into the matter and it was found that in such soils very little of the fluid really passed through the soil, but really passed around it and followed the filter paper along the side of the funnel. Hence the fact that the ammonia came through colorless, or nearly so, could not be relied upon to indicate that all the humus was removed. This apparatus reduces the labor to a minimum and permits many determinations to be carried on at once. The results appear in the following tables:

Index to soil samples.

No. 1. Peat soil, unproductive ("bogus").

No. 2. Peat subsoil, unproductive.

No. 3. Peat soil, productive.

No. 4. Peat subsoil, productive.

No. 5. Black soil containing sticky clay, unproductive ("bogus").

No. 6. Soil from Indiana Experiment Station plats, dark, compact clay loam, containing little sand; will not scour iron plow.

No. 7. The soil used in station lysimeters practically the same as No. 6.

No. 8. Clay soil from Allen County.

TABLE I.—*Comparison of the Grandeau method with the modified method (Huston's). Soils extracted with 2 per cent HCl. Ordinary filter used in Grandeau method.*

[5 grams soil used.]

Number of soil.	2 per cent ammonia.		4 per cent ammonia.		7.3 per cent ammonia.		8 per cent ammonia.	
	Grandeau.	Huston.	Grandeau.	Huston.	Grandeau.	Huston.	Grandeau.	Huston.
1 a	....	....	16.40	20.06	....	....	....	....
b	....	....	13.98	20.80	....	....	....	....
c	....	....	17.43	....	....	....	....	....
Mean	....	....	15.94	20.43	....	....	....	....
2 a	....	....	13.98	19.38	....	....	....	....
b	....	....	13.85	20.30	....	....	....	....
Mean	....	....	13.92	19.84	....	....	....	....
3 a	9.05	15.60	14.71	21.24	19.77	21.70	16.05	21.42
b	10.27	15.88	15.34	20.20	19.85	21.90	15.40	21.80
Mean	9.61	15.74	15.03	20.72	19.81	21.80	15.73	21.61
4 a	....	....	16.75	24.34	....	....	....	....
b	....	....	18.60	23.52	....	....	....	....
Mean	....	....	17.68	23.93	....	....	....	....
5 a	3.90	6.90	4.42	7.42	....	....	....	....
b	3.88	7.00	4.20	6.98	....	....	....	....
Mean	3.89	6.95	4.31	7.20	....	....	....	....
6 a	1.86	4.20	2.40	4.26	2.14	4.02	1.85	4.12
b	1.76	4.36	2.48	4.28	2.13	4.48	1.90	4.40
Mean	1.81	4.28	2.44	4.27	2.14	4.25	1.88	4.26
7 a	1.90	4.12	1.80	4.12	....	....	....	....
b	1.61	4.22	1.95	4.04	....	....	....	....
Mean	1.75	4.17	1.88	4.08	....	....	....	....

TABLE II.—*Comparison of Grandeau method, using porcelain plates, with modified method, one-half of 1 per cent acid used. Automatic washing apparatus.*

[2½ grams soil used.]

Number of soil.	2 per cent ammonia.		4 per cent ammonia.		7.3 per cent ammonia.	
	Grandeau.	Huston.	Grandeau.	Huston.	Grandeau.	Huston.
3 a	18.08	20.75	18.75	23.68	20.00	30.86
b	13.48	21.00	19.30	22.80	20.10	30.20
Mean	15.78	20.87	19.02	23.24	20.05	30.53
5 a	3.98	7.70	(1)	7.00	(1)	10.40
b	3.58	6.80	....	8.00	....	10.00
Mean	3.78	7.25	....	7.50	....	10.20
6 a	2.54	4.68	3.20	4.88	3.24	7.40
b	2.36	4.74	2.94	4.20	2.16	6.72
Mean	2.45	4.71	3.07	4.54	2.70	7.06
8 a	0.88	2.10	1.04	2.00	1.12	3.20
b	0.80	2.00	1.04	1.68	1.60	3.28
Mean	0.84	2.05	1.04	1.84	1.36	3.24

[1] Ammonia failed to pass through the soil layer.

The results show that the modified method gives much higher results than the original method of Grandeau.

The residues from the extraction with 2 per cent ammonia by Grandeau's method (see Table II) were transferred to the cylinders and treated with 2 per cent ammonia to see if a further quantity of humus could be obtained. The results appear in Table III.

TABLE III.—*Supplemental extraction of residues from Grandeau's method.*

Number of soil.	Extracted by Grandeau's method 2 per cent. ammonia.	Extracted in cylinders from residue.	Sum of extracts.
3 a.................	18.08	5.04	23.12
b.................	13.48	8.60	22.08
Mean........	15.78	6.82	22.60
5 a.................	3.98	3.04	7.02
b.................	3.58	3.92	7.50
Mean........	3.78	3.48	7.26
6 a.................	2.54	2.20	4.74
b.................	2.36	1.76	4.12
Mean........	2.45	1.98	4.43
8 a.................	0.88	1.00	1.88
b.................	0.80	1.12	1.92
Mean.... ...	0.84	1.06	1.90

It will be seen that the sum of the extracts agrees very well with column 2 in Table II.

In Table I it will be noticed that on the soils not peat the strength of the ammonia makes practically no difference in the modified method and comparatively little difference on peat soil when the strength is 4 per cent or over. In the Grandeau method there are wide differences, although this difference is not proportionate to the strength of the ammonia.

In Table II considerable differences appear in both methods as the strength of the ammonia is changed. Subsequent investigation showed that this variation was due to changes of temperature taking place in the room where the work was done. These variations in temperature did not occur when the work reported in Table I was in progress. The effect of temperature variation would be less, in the work reported, on the determination of Grandeau's method, since that work was carried on during the day, while the work on the modified method always extended over one night and sometimes over two. The high temperatures to which the work was exposed occurred at night.

INFLUENCE OF VARYING THE TIME OF DIGESTION.

This is shown for the modified method in Table IV. It was intended to use columns 2 and 4 of Table II in this comparison, since these samples there reported stood thirty-six hours. But the discovery of the variations due to change of temperature prevents the use of this material, since the work was done under different temperature conditions. So only work that is strictly comparable is included in Table IV,

TABLE IV.—*Influence of time of digestion, modified method.*

[0.5 per cent acid; disk filters.]

Number of soil.	2 per cent ammonia.			4 per cent ammonia.		7.3 per cent ammonia.
	60 hours.	72 hours.	96 hours.	60 hours.	72 hours.	60 hours.
3 *a*	18.32	25.20	24.80	28.00	28.06	29.52
b	18.20	25.60	24.45	27.20	27.16	29.60
Mean	18.26	25.40	24.62	27.60	27.63	29.56
5 *a*	5.04	7.52	6.84	7.44	8.08	9.00
b	5.68	(¹)	7.16	8.20	8.28	8.05
Mean	5.36	7.52	7.00	7.82	8.18	8.52
6 *a*	3.60	4.60	4.42	4.80	4.40	5.68
b	3.76	4.32	4.44	4.60	4.00	5.60
Mean	3.68	4.46	4.43	4.70	4.20	5.64
8 *a*	1.74	1.68	1.68	2.20	2.00	2.56
b	2.20	1.56	(¹)	2.40	1.52	2.32
Mean	1.97	1.62	1.68	2.30	1.76	2.44

[1] Material lost.

TABLE IV.—*Influence of time of digestion, modified method*—Continued.

[2 per cent acid, 4 per cent ammonia; ordinary filters.]

Number of soil.	36 hours.	48 hours.	68 hours.	96 hours.
3 *a*	21.24	22.28	24.04	....
b	20.20	21.70	23.94	....
Mean	20.72	21.99	23.99	....
6 *a*	4.28	4.00	....	4.40
b	4.26	4.01	....	4.85
Mean	4.27	4.01	....	4.63

The most conspicuous feature of Table IV is the increase in humus dissolved from the peat soil in 60 and 72 hours by 2 per cent ammonia. Ammonia of this strength failed to completely extract peat soil in all the work. With 4 per cent of ammonia the addition of 12 hours to the time of extraction fails to show any increase. The table also brings out again the effect of change in strength of ammonia solution used. For practical analytical purposes I believe 36 hours' digestion with ammonia of 4 per cent or over is sufficient.

INFLUENCE OF TEMPERATURE OF DIGESTION.

During the progress of the work it became evident that the temperature at which the digestion was conducted was a matter of importance. Four determinations have been made which will illustrate the extent of this variation.

TABLE V.—*Time, 36 hours, modified method.*

Number of soil.	Temperature of digestion.	
	80° F.	50° F.
3....	28.70	20.70
5.................	7.52	6.22

It would seem necessary to adopt some standard temperature at which humus determination should be made.

IS IT POSSIBLE TO COMPLETE THE EXTRACTION BY GRANDEAU'S METHOD IN A REASONABLE TIME?

This was found to depend upon the kind of soil. With a soil like No. 6 or No. 8, using disk filters, the whole amount of extract could be removed in a few hours. With No. 5 the ammonia soon stopped up the filter so that complete extraction was impossible. When ordinary filters were used No. 5 gave solutions still colored after a week's treatment and with No. 3 the operation was continued 10 days. The results on No. 3 appear in Table VI. About 10 cc. of fluid were applied at a time and this allowed to drain out before applying the next portion of 10 cc.

TABLE VI.—*Long-continued extraction on soil 3.*

[2 per cent acid, ordinary filters; total time, 10 days; 4 per cent ammonia.]

Amount of ammonia used.		(a)	(b)	Mean.
First extract	750 cc.	16.90	18.96	17.93
Second extract	250 cc.	2.80	2.38	2.59
Third extract..............	250 cc.	1.77	1.10	1.44
Fourth extract............	250 cc.	1.34	1.30	1.32
Fifth extract..............	250 cc.	0.89	0.85	0.87
Sixth extract..............	250 cc.	1.41	1.65	1.63
Seventh extract...........	250 cc.	2.10	1.80	1.95
Eighth extract	250 cc.	0.67	0.65	0.66
Ninth extract	250 cc.	0.57	0.50	0.53
Total.................	2,750 cc.	28.45	29.19	28.82

At the end of the sixth extraction the soil was again treated with 2 per cent acid to see if more calcium could be extracted. The acid filtrate contained calcium, and the acid extraction was continued until it was removed from the soil. The soil was then washed with water and the extraction with ammonia continued. The results show that at the end of 10 days considerable humus was passing into solution. A Sunday intervened between the fifth and sixth extraction, and the increase in the amount of the sixth over the fifth extraction is considered due to this longer standing in contact with the residual ammonia in the soil and with access of the air.

In those soils which yielded their humus readily to ammonia by the Grandeau process—that is, those in which the extract reached a maximum in a comparatively short time, the total amount extracted by the Grandeau process was always considerably less than extracted by the modified method.

COMPARISON OF DUPLICATES BY BOTH METHODS.

An examination of the preceding tables will give an idea of the relative differences of duplicate determination by the two methods. A series of determination was made to further test this point, the conditions being as nearly alike as possible for all.

TABLE VII.—*Comparison of duplicates.*

[Acid, 0.5 per cent. Ammonia, 7.3 per cent. Time for modified, 36 hours; time for Grandeau method, until filtrate was colorless or until soil did not permit more ammonia to pass through. Disk filter used.]

Number of soil.	Huston.	Grandeau.
3 *a*	25.00	20.40
b	24.80	16.49
c	24.48	11.10
d	24.80	23.26
Mean	24.77	17.81
6 *a*	4.10	2.23
b	4.20	1.60
c	4.00	2.23
d	3.70	2.55
Mean	4.00	2.15
8 *a*	1.50	.88
b	1.60	.82
c	1.56	.75
d	1.72	.74
Mean	1.58	.80

Expressed in terms of the percentage of the total amount involved in the determination, the differences between the highest and the lowest are:

Number of soil.	Huston's method.	Grandeau's method.
	Per cent.	*Per cent.*
3	2.1	62.6
6	12.5	44.2
8	13.9	17.5

The percentages of difference between duplicates have also been figured for soil No. 3, with four different ammonia solutions, using ordinary filters, and also for soil No. 7. In these cases also the result is in favor of the modified method.

PHOSPHORIC ACID, POTASH, ETC., IN HUMUS.

Are the amounts of phosporic acid, potash, etc., found in the ash of the humus necessarily associated with the humus, or is their presence to be ascribed to the solvent action of the ammonia and to the changes due to the laws governing the absorptive properties of soils?

Fifty determinations of phosphoric acid were made in the ash of humus, and the results showed no relation between the amount of humus extracted from the soil and the amount of phosphoric acid present. With the same soil a high extraction of humus was often accompanied by a low extraction of phosphoric acid. It seems more reasonable to believe that the presence of phosphoric acid in the ash is due to the independent action of ammonia on phosphates of iron and alumina in the soil. The irregularities in the amounts of phosphoric acids dissolved from the same soil are to be ascribed to the fact that at the temperature at which humus determinations are made the ammonia would require a very long time to decompose the phosphates, and to the fact that the amounts of phosphates dissolved by ammonia vary largely with changes of temperature. Thirty potash determinations were made in the ash

of humus and the same irregularities found as in the case of the phosphoric acid determinations. With the same soil there was no relation between the amounts of humus extracted and the amounts of potash in the ash.

The laws governing the exchange in bases in soil absorption are sufficient to explain the phenomena. It is not to be understood that we hold that the amounts of phosphoric acid, potash, etc., contained in the humus ash have no agricultural significance. For we believe the contrary to be true. These substances happen to be extracted from soils by the same process as the humus; but it does not follow that they were associated with the humus before its extraction.

We believe that other methods are better adapted for determining the available phosphoric acid, potash, etc., in the soil than the method in use for determining humus.

Mr. VAN SLYKE. I move that the method for soil analysis stand as adopted last year.

Mr. HUSTON. I would simply call attention to the fact that a provisional method was adopted for 1892–'93. I think there is much good material in Mr. Hilgard's paper.

The PRESIDENT. Why not refer the matter to the reporter for next year for further consideration?

Mr. VAN SLYKE accepted the suggestion, and changed his motion accordingly.

The motion was carried.

Mr. DE ROODE. Do I understand that the method as now printed in our proceedings of last year is to continue over?

The PRESIDENT. Yes, simply as a provisional method for another year. It now stands as a provisional method, and would of course remain such under the motion.

The president called for the report on dairy products.

Mr. Winton presented the following report:

REPORT ON DAIRY PRODUCTS.

By A. L. WINTON.

The study of methods for the analysis of dairy products during the current year has been confined to the determination of solid and fat in milk and cheese, the special subjects investigated being as follows :

(1) The drying of cheese and ether extract from cheese in air and in hydrogen.

(2) The Babcock gravimetric method for the determination of solids and fat in milk—(drying with asbestos in a perforated metal cylinder).

(3) The determination of fat by extraction after drying in air or in hydrogen.

(4) The calculation of solids in milk from the fat and the specific gravity.

Early in January circular letters were sent out to chemists of the association outlining the work and asking coöperation. No attempt was made to distribute samples, as such samples could only be kept in proper condition by sterilizing or treating with antiseptics, and material thus prepared is scarcely suitable for the testing of methods. It was also believed that by leaving each worker to make his own selection of material more and better work would be accomplished. A study of methods can often be taken up in connection with regular station work and with little additional labor when opportunity might not be presented for a separate investigation on committee samples, and the conclusions drawn from results obtained on a variety of materi... are certainly of more value as illustrating the applicability

of methods than if a greater number of analyses were made on but two or three samples.

Reports were received from the following chemists:

B. W. Kilgore, North Carolina.
R. H. Smith, Massachusetts.
Charles Ryan, Colorado.
F. S. Shiver, South Carolina.
T. L. Lyon, Nebraska.
H. I. Patterson, Maryland.

C. L. Penny, Delaware.
R. E. Hardee, Georgia.
R. E. Blouin, Louisiana.
A. W. Ogden and A. C. Winton, Connecticut.

THE DRYING OF CHEESE AND OF ETHER EXTRACT FROM CHEESE, IN HYDROGEN AND IN AIR.

The report on dairy products for 1892 brought out clearly two points with regard to the determination of moisture in cheese, namely: (1) That the drying of cheese at 100° C. to constant weight is a slow and in some cases interminable process, and (2) that cheese loses about 1 per cent less when dried over sulphuric acid at ordinary temperatures than when dried in air at 100° C. These facts and others led the reporter (Mr. Babcock) to the conclusion that matters other than water are driven off at the temperature of boiling water. With regard to drying ether extract, Mr. Babcock writes: "I find that where cheese has been dried at ordinary temperatures that it requires a very long time for the ether extract to be dried to constant weight,—almost as long, indeed, as it did to dry the cheese at 100° to constant weight, seeming to indicate that there are volatile matters which are soluble in ether."

If the matters other than moisture which are removed from cheese and ether extract at 100° C. exist already formed in the cheese, it would seem impossible to so conduct the drying at that temperature as to avoid their loss; if, however, they are formed by oxidation during the heating in the air, a remedy should be found in drying in hydrogen. Your reporter, therefore, in accordance with instructions and with the coöperation of other chemists, undertook a comparison of drying both cheese and ether extract from cheese in air and in hydrogen.

It was thought advisable to dry ether extract in a current of air as well as in the flask open to the air of the cell in order to insure complete removal of ether and secure the best conditions for oxidation.

Those taking part in the work were requested to make their tests on several varieties of cheese, taking especial care in the preparation of the sample.

Mr. R. H. Smith reports analyses of five samples—three of domestic full cream cheese, one of imported *Fromage de Roquefort* and one of imported *Crème de Brie*.

The following table shows the percentage loss by drying at 100° C. in hydrogen for periods of four hours, and by drying in air to constant weight:

Time of heating (periods of 4 hours).	Full cream cheese.			Fromage de Roquefort.	Crème de Brie.	Average.
	Sample No. 1.	Sample No. 2.	Sample No. 3.			
Dried in hydrogen:	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
First period	39.46	38.78	35.55	37.92	52.54	....
Second period	.24	.17	.11	.42	.18	....
Third period............	.09	.16	.10	.09	.12	....
Fourth period	.09	....	.03	.11	.07	....
Fifth period	.06	....	.02	.04	.08	....
Sixth period............	....	....	....	.06	.07	....
Seventh period	....	....	....	.02	.09	....
Eighth period	....	....	....	....	.07	....
Total loss dried in hydrogen............	39.94	39.11	35.81	38.66	53.22	41.35
Total loss dried in air..	39.81	38.72	35.87	38.16	52.57	41.03

By drying ether extract in air and in hydrogen Mr. Smith obtained the following results:

	Dried in hydrogen current.	Dried in air current.	Dried in air (open flask).
	Per cent.	*Per cent.*	*Per cent.*
Full cream cheese (first lot):			
First extraction (2 hours)	33.89	33.94	34.68
Second extraction (2 hours)	.06	....	.15
Total per cent fat	33.95	33.94	34.83
Full cream cheese (second lot):			
First extraction (2 hours)	32.08	32.11	33.17
Second extraction (2 hours)	.17	.13	.09
Total per cent fat	32.25	32.24	33.26
Fromage de Roquefort			
First extraction (2 hours)	33.03	32.86	33.64
Second extraction (2 hours)	.03	.18	.05
Third extraction (2 hours)	.07	....	....
Fourth extraction (2 hours)	.07	....	....
Total per cent fat	33.20	33.04	33.69
Crème de Brie:			
First extraction (2 hours)	25.24	24.90	24.93
Second extraction (2 hours)	....	.06	....
Total per cent fat	25.24	24.96	24.93

Referring to Mr. Smith's analyses, Mr. J. B. Lindsay writes:

"It would appear from our figures that with the cheese used by us a current of air gave practically the same results as hydrogen. I suppose that hydrogen would be on some accounts a trifle safer; still, air is cheaper, and if others get like results with ourselves it would indicate that it might be left to the discretion of the workers to use air or hydrogen, as most convenient. Drying in dead air seems to give slightly different results, which is not to be wondered at."

Mr. T. L. Lyon gives his results as follows:

Determination of moisture: Per cent.

By drying to constant weight in a current of hydrogen gas at the temperature of boiling water 37.84

By drying to constant weight in an open dish at the temperature of boiling water 38.39

Drying ether extract:

Portions of cheese were extracted by Short's method, the extract evaporated nearly to dryness and then dried, No. 1 in a current of air, No. 2 in a current of hydrogen. The percentage weight of the dried extract was—

No. 1 .... 28.46

No. 2 .... 28.61

Mr. R. E. Hardee describes the material used in his experiments as follows:

"This cheese was one year and one month old when taken for analysis. Our dairyman made it for a 'long keeper,' so as to try the effect of a Georgia summer on it. Less rennet, more salt, and a higher scald were used than usual. The taste was a little old."

The results obtained by Mr. Hardee on this cheese are as follows:

Loss at 96° C.

Periods of heating.	Air.			Hydrogen.	
	I.	II.	III.	I.	.II.
First	17.63	17.79	17.11	14.87	14.21
Second	.48	.32	.43	.71	1.39
Third......................	.21	.21	.12	.42	.40
Fourth	.07	.09	.04	.23	.47
Fifth	.07	.01	.01	.10	.13
Total loss.............	18.46	18.42	17.71	16.33	16.60
Average...................	18.44[1]		....	16.46	

[1] III not included.

The periods of heating varied from 2½ to 4 hours.

Fat.

Time of drying of fat at 96° C.	5 grams cheese, 10 grams $CuSO_4$ fat dried in—				5 grams cheese, 5 grams $CuSO_4$ fat dried in air.
	Air.		Hydrogen.		
Two hours......................	——	43.01	43.04	43.03	43.45
Four hours	42.96	42.88	42.73	42.73	43.35
Six hours......................	42.96	42.88	42.72	42.70	43.35
Average after 6 hours	42.92		42.70		43.35

Mr. Charles Ryan reports his results on a single sample of cheese made at the Fort Collins (Colo.) factory:

Loss at 95° C.—Portions of about 5 grains each were dried in tubes with asbestos at the temperature of boiling water, which at the elevation of the Colorado station is 95° C. An apparatus designed for drying fodders was used, some of the tubes being connected with a hydrogen supply, others being left open to the air. In the latter case the escape of moisture was, of course, slow. The results follow:

Period of heating (4 hours).	Air.			Hydrogen.			
	I.	II.	III.	I.	II.	III.	IV.
First......................	20.00	10.17	14.21	36.13	37.33	34.15	37.32
Second.....................	7.50	13.88	11.40	1.44	1.84	2.77	1.08
Third......................	3.16	4.53	4.09	.23	.32	.27	.20
Fourth	3.06	4.96	4.01	.26	.08	.32	.47
Fifth	1.59	1.35	1.54	.30	.39	.22	.19
Total loss in 20 hours..	35.31	34.89	35.25	38.36	39.96	37.73	39.26

As the drying in the open tubes was not carried to completion, the results are not comparable with those dried in hydrogen to practically constant weight. The results by the latter method show that drying in hydrogen, like drying in air, is a slow process, sixteen hours not being sufficient to secure constant weight.

Fat.—"Five grams of the cheese were weighed out and finely pulverized with anhydrous copper sulphate, then placed in a glass cylinder and dried in the hot-air bath for one hour at 100° C. The cylinder was then tranferred to a Soxhlet apparatus and

the fat extracted with anhydrous ether for fifteen hours." Three extracts obtained by this method were dried at 95° C., with the following results:

Time of drying.	Dried in air current.		Dried in hydrogen current.		Dried in air (open dish).	
	I.	II.	I.	II.	I.	II.
2 hours......................	34.41	33.36	34.07	33.23	32.60	32.28
4 hours......................	34.37	33.36	34.05	33.16	32.42	32.28
Average, 4 hours............	33.86		33.60		32.35	

Mr. H. J. Patterson sends the following methods of preparation of samples: Samples Nos. 1 to 4 were full cream cheese; sample No. 5, Neufchatel; (a) cutting sample in very fine pieces A. O. A. C.; (b) cutting fine and mixing with anhydrous $CuSO_4$—A. O. A. C.; (c) taking 1 part cheese, 2 parts quartz powder and thoroughly pulverizing in mortar until the mass is uniform and granular.

The various determinations were made after the A. O. A. C. methods and following the instructions sent out by the reporter. When the results of duplicates were within 0.2 per cent of each other, the average is given; when more, both results are given.

Moisture—water oven 99°.

Sample No.	Drying.	Sample mixed with silica powder.	Sample cut very fine.
	Hours.	Per cent.	Per cent.
No. 1......................	4	29.15	32.21
	8	29.23	33.37
	12	29.30	33.66
No. 2......................	4	26.75	30.67
	8	26.92	31.98
	12	26.85	32.24
No. 3......................	4	28.22	27.64
	8	28.22	29.18
	12	28.20	29.52
No. 4......................	4	25.67	{ 24.51 { 25.03
	8	25.62	26.76
	12	25.65	27.15
No. 5......................	4	63.07	65.22
	8	62.98	65.89
	12	63.10	65.99

Ether extract.

	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Sample No. 5.
Cheese pulverized with quartz powder:					
Dried in water oven—					
1 hour					8.22
2 hours	34.35	35.47	39.30	39.60	8.05
3 hours					8.10
4 hours					8.05
Dried in water oven in current of air—					
1 hour	33.80 / 34.75				7.92
2 hours	33.40 / 34.20				7.88
3 hours	33.55 / 34.35				7.88
Dried in water oven in current of hydrogen—					
1 hour	33.95				7.85
2 hours	33.40				
3 hours	33.70				7.55
4 hours	33.45				7.60
Cheese mixed with anhydrous $CuSO_4$. Short:					
Dried in water oven—					
1 hour		34.70			9.29 / 8.94
2 hours	34.32	34.54	40.76	40.47	9.17 / 8.82
3 hours		34.52			9.14 / 8.75
4 hours		34.48	40.69	40.39	9.11 / 8.72
5 hours		34.48			9.05 / 8.65
6 hours				40.33	
8 hours				40.29	
Cheese cut very fine and extracted:					
Dried in water oven—					
1 hour					7.93 / 8.45
2 hours	32.02	27.64 / 30.49	38.61 / 39.94	40.54 / 39.79	7.86 / 8.36
3 hours					7.86 / 8.33
4 hours		27.34 / 30.48		40.30 / 39.77	7.84 / 8.31
6 hours		27.64 / 30.48		40.30 / 39.77	7.81 / 8.25

By the use of quartz powder the cheese may be thoroughly mixed and a correct sample obtained, and this seems to be shown by the more uniform results obtained; also, by this means we may procure a sample that can be handled with ease.

After being mixed the sample is much like sugar in appearance.

The samples used in my own experiments were two—one a full-cream cheese about four months old, the other a hard cheese, resembling somewhat Edam, made in West Burlington, N. Y.

Loss at 100°.—Duplicate portions of 5 grams each were dried in open dishes in the air bath and in flask, passing a current of hydrogen in and out through a double-bored stopper during the drying. The results follow:

Time of heating (period of 4 hours).	Full-cream cheese.		Hard cheese.	
	Air.	Hydrogen.	Air.	Hydrogen.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
First period	37.22	38.36	17.48	16.72
Second period	.69	.13	.35	.94
Third period.................	.12	.00	.28	.41
Fourth period	.05	....	.12	.03
Fifth period	.00	....	.04	.12
Sixth period	....	....	.12	.09
Seventh period	....	....	.05	.04
Eighth and ninth periods...	....	....	.09	.11
Tenth and eleventh periods.	....	....	.13	.11
Total loss	38.08	38.49	18.66	18.57

Fat.—Three extracts were obtained by Short's method. The first was dried in an open flask exposed to the air of the cell, the second in a current of hydrogen, and the third in a current of air. Following are the results:

Time of drying.	Full-cream cheese. fat dried in—			Hard cheese, fat dried in—		
	Air current.	Air, open flask.	Hydrogen current.	Air current.	Air, open flask.	Hydrogen current.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Two hours	32.09	32.31	31.96	41.60	42.79	41.25
Four hours	32.02	32.07	31.90	41.52	41.41	41.24
Six hours	32.01	32.02	31.90	41.52	41.41	41.24

A summary of the results of the different analyses is given in Table I.

TABLE I.—*Comparison of the results of drying cheese and ether extract in air and in hydrogen.*

Analyst.	Loss at 100°.			Fat.			
	Number of samples.	Air.	Hydrogen.	Number of analyses.	Dried in air (open flask).	Dried in air current.	Dried in hydrogen current.
		Per cent.	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
R. H. Smith.....................	5	41.03	41.35	4	31.68	31.05	31.16
T. L. Lyon	1	38.39	37.84	1	....	28.46	28.61
R. E. Hardee....................	1	18.44	16.46	1	42.92	....	42.70
Chas. Ryan	......	....	....	2	32.35	33.86	33.60
H. J. Patterson	......	....	....	2	21.20	20.91	20.52
A. L. Winton	2	28.37	28.53	2	36.71	36.76	36.57
All samples.................	9	35.41	35.35	11	....	30.52	30.42

The differences in the results on loss at 100° by the two methods are not, as a rule, marked, and can generally be attributed to lack of uniformity in the sample. In some cases hydrogen gave the higher results, in others air, the average of all the results by each of the two methods being practically identical. Mr. Hardee, how-

ever, working on a sample of old cheese with a low water content, found that hydrogen drying gave results about 2 per cent low. If the results of the analysis of this sample are excluded, the average results for drying in air and hydrogen would be 37.53 and 37.70, respectively. It is evident that there is no such regular difference between drying at 100° in air and in hydrogen as between drying at 100° in air and at ordinary temperatures, over sulphuric acid.

The point which is brought out clearly by the work on cheese is that a gradual loss in weight takes place at 100° C. in hydrogen, just as in air, indicating that if volatile organic constituents are driven off at that temperature in the latter case they are in the former. Both methods being at fault, and drying over sulphuric acid being altogether too tedious an operation for ordinary work, there is every reason for adopting a simple conventional method such as is given in the proceedings of this association for 1892, p. 224, viz, drying at 100° C. for ten hours.

On referring to the results on fat, it will be seen that drying the ether extract in hydrogen gave results on an average 0.11 per cent lower than drying in a current of air, but this difference is slight, considering the errors of sampling and the high per cent of fat. In some instances drying in an open flask gave results much too high, due, probably, to the incomplete removal of ether. This source of error can be easily avoided by occasionally exhausting the air from the flasks during the drying, by means of a hand bellows.

Although our method for the proximate analysis of cheese yields results which represent approximately the truth and are much more satisfactory than the methods for fodder analysis, still they can not boast of very great accuracy. The errors of water determination have been considered. Ether extract contains, in addition to pure fats, cholesterin, free lactic and butyric acids, and other impurities. But the chief error, in my opinion, is occasioned by the use of an improper factor for the calculation of casein from the nitrogen.

Chittenden and Painter[1] find 15.91 per cent and Hammersten[2] 15.65 per cent of nitrogen in the casein of milk; which would make the factor for pure casein 6.28 and 6.39, respectively—average 6.33.

But according to Hammersten[3] the proteid of cheese is not identical with the casein of milk, the latter being split up through the action of rennet into two proteids, one of which is precipitated, the other, of which there is relatively but a small quantity, remains in solution. Almost all the nitrogenous matter of fresh cheese consists of the precipitated proteid to which Schulze has given the name "paracasein," and which according to analyses by Roese and Schulze contains 15.14 per cent of nitrogen.[4] The factor for "paracasein" is, then, 6.60.

Ripened cheese contains not only "paracasein," but decomposition products of this proteid which vary in quantity and character with the kind and age of the cheese. Benecke and Schulze[5] found in Emmenthal and various other Swiss cheeses, in more or less ripened condition, that from 5 per cent to 27 per cent of the nitrogenous matter consisted of leucin, tyrosin, glutamic acid (all of which contain a much lower percentage of nitrogen than "paracasein"), with some ammonia and other decomposition products. Some cheeses, Camembert, for example, smell strongly of ammonia, showing that the chemical changes which take place are accompanied by loss of nitrogen. It is evident, then, that the factor 6.25 is too low, both for fresh and for ripened cheese. The per cent of undetermined matters obtained by difference, which includes a small amount of organic acids, represents, in all probability, largely error, occasioned by the use of the factor 6.25. I am inclined to believe that our analyses would represent more nearly the facts, if crude protein were obtained by difference, substracting the sum of the water, ash, and fat from 100.

[1] Studies from the Laboratory of Physiological Chem., Yale Univ., 2, 172.
[2] Ztschr. für physiolog. Chem., 7, 269.
[3] Jahr. für Thierchemie 1874 135., Land. Jahrbächer 1887, 16, 319.
[4] Land. Versuchs. 31, 115.
[5] Land Jahr. 16, 319.

THE BABCOCK GRAVIMETRIC METHOD FOR THE DETERMINATION OF SOLIDS AND FAT IN MILK (DRYING WITH ASBESTOS IN A PERFORATED METAL CYLINDER).

The value of asbestos as an absorbent in milk analysis has become quite generally recognized, and a number of asbestos methods have been devised.

In the "alternate method," adopted by this association some years since (see Proceedings for 1892, p. 223), the milk is dried with asbestos in a glass tube, air being drawn through the tube during the drying. This method is both rapid and accurate; the apparatus is, however, somewhat complicated.

At some stations milk is dried in a current of hydrogen in an apparatus, such as is used for the determination of water in fodders. While it is true that this method is theoretically the nearest perfect, still numerous experiments (including some reported herewith) indicate that practically the same results are obtained, at least on fat, when the drying is done in air.

Mr. J. M. Bartlett describes the method used at the Maine station as follows: "We use asbestos on tin foil placed in a 2½-inch watch glass for drying, and then this foil is drawn up over the asbestos, perforated with a pin, and placed in the extractor for determination of fat. The watch glass is provided with a cover and clamp, so a desiccator is not necessary."

At the North Carolina station asbestos is used in a double-stoppered glass tube, the drying and extracting being carried on in the usual manner.

The Babcock perforated-cylinder method has been in use at the Wisconsin station for over four years, and at the Connecticut station and other stations nearly as long, as it is believed to have advantages over other methods which recommend it to the association. The plan for testing this method is given in the following extract from my circular letter:

Compare the method here described with your standard method:

"Provide a hollow cylinder of perforated sheet metal 60 mm. long and 20 mm. in diameter, closed 5 mm. from one end by a disk of the same material. The perforations should be about 0.7 mm in diameter and as close together as possible. Fill loosely with 1.5 to 2.5 g of dry woolly asbestos and weigh. Introduce a weighed quantity of milk (about 5 grams). Dry at 100° C. for four hours. During the first part of the drying the door of the cell should be left partly open to allow escape of moisture. Cool in a desiccator and weigh. Repeat the drying until the weight remains constant. Extract with anhydrous ether for two hours. Evaporate off the ether and dry the fat at 100° C. Weigh. Repeat the extraction until nothing further is removed. It would also be interesting to determine the fat by difference, drying the extracted cylinders at 100°."

To such chemists as expressed a willingness to take part in the work I sent one of the perforated metal cylinders, together with a sample of asbestos suitable for the method. The cylinders distributed were made of perforated tin plate. I have since been able to obtain perforated sheet brass, and prefer this material to tin, which is liable to rust on exposure to the fumes of the laboratory.

The following extracts are from the reports of the analysts who took part in the work:

Mr. J. B. Lindsay, of the Massachusetts station, writes:

"I send you the results of our analyses of milk and cheese, according to methods described in your circular of the past winter. The analytical work has been carried out by Mr. R. H. Smith, of this station. The morning's and evening's milk, mixed, of six cows belonging to the station was tested, using the Babcock method, as described in your circular, and the sand method in general use at this station. I would say, relative to the Babcock method, that the tube containing the asbestos was heated for two hours in the drying bath, cooled, and weighed; about 5 cc. of milk were then added, the whole accurately weighed and dried, as per instructions, for four hours. Our drying bath has a constant temperature of 102° to 104° C. After the first drying of four hours no further loss was noted in case of total solids. In case of fat,

the second extraction of two hours gave an average increase of 0.08 per cent. The ether used was Squibb's best, and not strictly anhydrous.

"The sand method is old, and generally known. A quantity of acid-washed sand was brought into a small porcelain dish, dried for an hour or two, cooled, and weighed. Five cubic centimeters of milk were then added, the dish and its contents again accurately weighed, and dried five or six hours to a constant weight. One drying is generally sufficient. The sand is then brought into a porcelain mortar, rubbed fine, placed upon a filter, and the fat worked out with Squibb's ether, the ether evaporated, and the fat dried in the drying cell at 102° to 104° C. to constant weight.

"I was much pleased with the Babcock method, and see no reason why it is not a perfectly safe and reliable method."

Mr. B. W. Kilgore reports as follows:

"The gravimetric method for fat in milk in use in this laboratory is very similar to the one described in your circular letter, except we use a double-stoppered glass tube for drying and extracting, instead of the perforated metal cylinder, asbestos being used as the absorbent. We usually extract three to four hours, using about 5 cc. of milk.

"Total solids were determined by drying four to six hours in air bath at 100° C. in the perforated metal cylinder and in the glass tube on asbestos and in open platinum dish on sand. The results are in all cases somewhat lower by the six than the four hours' drying. The solids obtained by drying in an open platinum dish are slightly higher than those in the perforated-cylinder and glass-tube methods. This is likely due to some evaporation in the former case, since the milk was measured into the dish and it was weighed without cover."

Mr. David O'Brine, of the Colorado station, writes: "My assistant [Mr. Ryan] did the work, and you will please give him credit for it."

Mr. Ryan gives results by the cylinder and the Adams coil methods. In both cases about 5 grams of milk were dried to constant weight at 100° C. and extracted in Soxhlet's apparatus. The percentages of fat for successive extractions are given, as follows:

	2 hours.	4 hours.	6 hours.
	Per cent.	Per cent.	Per cent.
Sample No. 1, Babcock	4.29	4.33	4.35
Babcock	4.26	4.28	4.28
Adams	4.16	4.28	4.28
Sample No. 2, Babcock	3.91	4.00	4.04
Babcock	3.89	3.99	4.00
Adams	3.76	3.84	3.84
Sample No. 3, Babcock	3.97	3.97	3.97
Adams	4.02	4.08	4.08

Solids were also determined by drying at 110° C. with sand and alone in a platinum dish.

The following extracts are taken from Mr. F. S. Shiver's report:

"All were samples of whole milk, except one, which was a sample of sweet milk, from which the cream had been partially removed. Total solids and fat were determined in the cylinder, as directed in your circular letter. For comparison total solids were determined in some cases by drying on asbestos, on sand, and in a dish alone without any absorbent. Fat was determined by extracting the total solids on asbestos in some cases, and in others by extracting the total solids on sand, with water-free ether. Five hours at boiling-point of water was found sufficient for complete desiccation of total solids and an extraction of eight hours in the case of

fat was found sufficient. In the case of the cylinder four hours were sufficient for complete desiccation of total solids, the temperature of boiling water being employed. Fat was practically all removed in two hours' extraction as recommended in your circular letter; further extraction of two hours gave practically no increase in per cent. Water-free ether was used in the extraction."

Mr. B. B. Ross writes: "The Babcock perforated cylinder has given most satisfactory results. I give the following as the result of a comparative test with the Adams' method:

	Per cent fat.
By Adams' process	4. 11
By perforated tube, with asbestos	4. 09
By difference in later case	4. 00

"The tests were made by Mr. R. E. Blouin."

Mr. H. H. Nicholson, of Nebraska, writes that the work was done for him by Mr. Lyon. "Cylinders were dried in air bath at the temperature of boiling water. For a comparison the Caldwell drying tube, filled with ignited asbestos, was used, and the milk dried in a current of hydrogen at the same temperature. Both were extracted in the same manner."

At the Connecticut State station four samples of herd's milk were analyzed by several methods by A. W. Ogden and myself. The figures reported are in each case the average of closely agreeing duplicates, one by each chemist. The asbestos used in the cylinders was ignited previous to use. Four hours drying, at 100° C. was generally found sufficient for solids, although in some cases an additional heating of four hours lowered the results by about 0. 05 per cent. Fat was practically all removed in two hours' extraction. The largest increase for a subsequent extraction of two hours was 0. 03 per cent. It is not certain, however, that two hours' extraction would be sufficient under all circumstances; the figures reported merely show that the fat is easily and quickly removed by ether.

Very good results on fat were obtained by the metal-cylinder method, drying the residue after extraction, weighing and calculating the fat by difference. It was found that a very thorough drying was necessary in order to secure good results. This indirect method would seem worthy of further trial, and if found efficient would furnish a valuable check for the direct method.

In Table II are given the results by the different chemists on fat and solids in milk, cream, skim milk and buttermilk, also solids calculated by formulæ to which reference will be made later.

The average results on fat in whole milk, comparing the perforated-cylinder method with other methods, are given in Table III.

It will be noted that as a rule the agreement on fat is remarkably close, the average results by the perforated-cylinder method being slightly higher than by the other methods. The greatest average difference was between the cylinder and other asbestos methods where theoretically the results should have been identical, indicating that the slight difference was due to analytical error.

Table II.—*Comparative results on fat and total solids in milk, cream, etc.*

| | | Fat. | | | | | | | |
| | | Milk dried on asbestos in— | | | | | | | |
	Specific gravity.	Perforated metal cylinder (fat weighed).	Perforated metal cylinder (fat by difference).	Glass tube, air.	Dish.	Glass tube, hydrogen (fat weighed).	Glass tube (fat by difference).	Sand method.	Paper-coil method.
R. H. Smith (Massachusetts):		*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Milk from cow No. 1	....	4.74	4.63	....	....	....	....	4.62	....
Milk from cow No. 2	....	3.61	3.72	....	....	....	....	3.59	....
Milk from cow No. 3	....	4.09	3.93	....	....	....	....	4.14	....
Milk from cow No. 4	....	4.21	4.17	....	....	....	....	4.16	....
Milk from cow No. 5	1.0329	4.15	4.24	....	....	....	....	4.17	....
Milk from cow No. 6	1.0320	4.76	4.80	....	....	....	....	4.61	....
B. W. Kilgore (North Carolina):									
Whole milk	1.0299	4.36	....	4.31	....	....	....	....	....
Skim milk	1.0350	....	....	.88	....	....	....	....	....
Cream.....................	1.0000	21.63	....	21.56	....	....	....	....	....
Whole milk	1.0330	....	....	4.15	....	....	....	....	....
Skim milk....................	....	....	....	.73	....	....	....	....	....
Cream.....................	....	20.48	....	20.49	....	....	....	....	....
Whole milk	1.0340	4.46	....	4.35	....	....	....	....	....
Cream.....................	....	....	....	19.87	....	....	....	....	....
Buttermilk	1.0340	1.02	....	1.01	....	....	....	....	....
Skim milk	1.0390	....	....	.35	....	....	....	....	....
Whole milk	1.0340	4.50	....	4.40	....	....	....	....	....
Cream.....................	1.0190	....	....	18.99	....	....	....	....	....
Charles Ryan (Colorado):									
Milk from short-horn cow.....	....	4.30	....	....	....	....	....	....	4.28
Do	....	4.02	....	....	....	....	....	....	3.84
Do	....	3.97	....	....	....	....	....	....	4.08
F. S. Shiver (South Carolina):									
Whole milk from grade Jersey.	1.0261	4.43	....	....	....	....	....	4.40	....
Do	1.0290	3.22	....	....	3.16	....	....	....	....
Whole milk from herd of Jerseys	1.0323	....	....	....	4.08	....	....	....	....
Milk, partly skimmed, from grade Jersey..................	1.0285	1.95	....	....	....	....	....	....	....
Whole milk from herd of Jerseys	1.0299	....	....	....	4.89	....	....	4.86	....
Whole milk from herd of Holsteins........................	1.0299	3.27	....	....	....	....	....	3.21	....
T. L. Lyon (Nebraska):									
Whole milk.....................	....	4.00	....	....	....	4.06	....	....	....
A. W. Ogden and A. L. Winton (Connecticut):									
Herd's milk....................	1.0345	3.11	3.00	....	....	3.06	....	3.03	....
Do	1.0339	2.72	2.68	....	....	2.69	2.66	2.69	....
Do	1.0337	4.12	4.12	....	....	4.09	4.05	4.04	....
Do	1.0324	4.38	4.30	....	....	4.28	4.20	4.40	....

TABLE II.—*Comparative results on fat and total solids in milk, cream, etc.*—Continued.

	Total solids.							Total solids calculated.		
	Milk dried on asbestos in—									
	Metal cylinder.	Glass tube, air.	Dish.	Glass tube, hydrogen.	Sand method.	Paper-coil method.	Milk dried alone in dish.	Babcock.	Hehner & Richmond.	Fleischmann.
R. H. Smith (Massachusetts):	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.
Milk from cow No. 1....	13.93	......	......	......	14.03	......	......	......	......	......
Milk from cow No. 2....	12.57	......	......	......	12.66	......	......	......	......	......
Milk from cow No. 3....	13.77	......	......	......	13.87	......	......	......	......	......
Milk from cow No. 4....	13.14	......	......	......	13.21	......	......	......	......	......
Milk from cow No. 5....	13.55	......	......	......	13.56	......	......	13.59	13.23	13.47
Milk from cow No. 6....	13.53	......	......	......	13.59	......	......	14.04	13.62	13.92
B. W. Kilgore (North Carolina):										
Whole milk..............	12.81	......	......	......	......	......	......	13.04	12.66	12.93
Skim milk	......	10.23	......	......	......	......	......	10.16	9.96	10.06
Cream	28.72	28.72	......	......	......	......	......	......	......	......
Whole milk..............	......	13.31	......	......	......	......	......	13.61	13.23	13.50
Skim milk	......	9.66	......	......	10.37	.....	......	......	......	......
Cream	27.80	28.05	......	......	23.36	......	......	......	......	......
Whole milk..............	13.55	13.52	......	......	13.74	......	......	14.18	13.75	14.05
Cream	......	26.45	......	......	27.17	......	......	......	......	......
Buttermilk	9.25	9.12	......	......	9.29	......	......	10.06	9.86	9.98
Skim milk	......	9.58	......	......	9.98	......	......	10.22	10.36	10.41
Whole milk..............	13.57	13.51	......	......	13.77	......	......	14.24	13.81	14.11
Cream	......	26.87	......	......	27.02	......	......	27.92	26.94	27.77
Charles Ryan (Colorado):										
Milk from short-horn cow	13.59	......	......	......	......	10.72	......	......	......	......
Do.................	13.67	......	......	......	13.99	10.70	13.55	13.80	13.42	......
Do.................	14.14	......	......	......	......	11.48	......	......	......	......
F. S. Shiver (South Carolina):										
Whole milk from grade Jersey..................	12.04	......	12.21	......	12.08	......	......	12.13	11.78	12.08
Do.................	10.99	......	11.13	......	......	......	11.20	11.40	11.08	11.34
Whole milk from herd of Jerseys..................	......	......	13.15	......	......	......	13.23	13.31	12.95	13.26
Milk, partly skimmed, from grade Jersey	9.68	......	9.88	......	......	......	9.81	9.78	9.54	9.72
Whole milk from herd of Jerseys..................	......	......	13.60	......	13.46	......	......	13.68	13.27	13.59
Whole milk from herd of Holsteins..............	11.26	......	11.21	......	11.26	......	......	11.69	11.37	11.63
T. L. Lyon (Nebraska):										
Whole milk.............	13.39	......	......	13.21	......	......	......	......	......	......
A. W. Ogden and A. L. Winton (Connecticut):										
Herd's milk	12.38	......	......	12.20	12.25	......	......	12.66	12.35	12.53
Do.................	11.93	......	......	11.83	11.78	......	......	12.08	11.78	11.97
Do.................	13.67	......	......	13.51	13.54	......	13.57	13.72	13.30	13.59
Do.................	13.55	......	......	13.39	13.47	......	13.47	13.65	13.25	13.53

TABLE III.—*Comparison of average results on fat in whole milk by the perforated-cylinder method and other methods.*

Analyst.	Number samples.	Perforated cylinder (asbestos).		Other asbestos methods (milk dried in air).	Milk dried in hydrogen (asbestos).	Sand.	Paper coil.
		Fat weighed.	Fat by difference.				
		Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	
R. H. Smith...............	6	4.26	4.25	....	....	4.22	....
B. W. Kilgore...............	3	4.44	....	4.35	....	....	....
Chas. Ryan...............	3	4.10	....	....	....	....	4.06
F. S. Shiver...............	2	3.85	....	....	....	3.80	....
Do...............	1	3.22	....	3.16	....	....	....
T. L. Lyon	1	4.00	....	....	4.06	....	....
R. E. Blouin...............	1	4.09	4.00	....	....	....	4.11
A. W. Ogden and A. L. Winton...............	4	3.58	3.52	....	3.52	3.54	....
All analysts	11	3.99	3.96	....	....	....	....
	4	4.13	....	4.05	....	....	....
	5	3.65	....	....	3.63	....	....
	12	3.96	....	....	....	3.92	....
	4	4.10	....	....	....	....	4.07
Difference compared with the cylinder method	....	....	—.03	—.08	—.02	—.04	—.03

A comparison of the average results on total solids in whole milk is given in Table IV. When asbestos or sand were used as absorbents, or when the milk was dried alone in a dish, the results generally agree closely with each other.

Compared with the cylinder method the results by other asbestos methods and the sand method were in both cases on an average 0.04 per cent high, while drying the milk alone in a dish gave results 0.02 per cent low.

In every case drying in hydrogen gave a slightly lower result than drying in air, the average difference being 0.15 per cent. These results are in harmony with those of Schmöger, who obtained an average of about 0.10 per cent less solids by drying in hydrogen than by the sand method.

The accuracy of the method for the analysis of cream, skim milk, and buttermilk is illustrated by some results given in Table II.

TABLE IV.—*Comparison of average results on total solids in whole milk by the perforated-cylinder method and other methods.*

Analyst.	Number samples.	Perforated cylinder (asbestos).	Other asbestos methods (milk dried in air).	Milk dried in hydrogen (asbestos).	Sand.	Paper coil.	Milk dried alone.
		Per cent.	Per cent.	Per cent.	Pr cent.	Pr cent.	Pr cent.
R. H. Smith	6	13.42	....	....	13.48	....	....
B. W. Kilgore	2	13.56	13.52	....	13.75	....	....
Chas. Ryan...............	1	13.67	....	....	13.99	10.70	13.55
F. S. Shiver	2	11.65	11.71	....	11.67	....	....
Do...............	1	10.99	11.13	....	....	....	11.20
T. L. Lyon	1	13.39	....	13.21	....	....	....
A. W. Ogden and A. L. Winton	2	13.61	....	13.45	13.50	....	13.52
Do...............	2	12.15	....	12.02	12.02	....	....
All analysts	5	12.28	12.32	....	....	....	....
	5	12.98	....	12.83	....	....	....
	15	13.08	....	....	13.12	....	....
	4	12.97	....	....	....	....	12.95
Difference compared with the cylinder method	....	....	+.04	—.15	+.04	....	—.02

The results which have been reported demonstrate the accuracy of the perforated-cylinder method for the determination of both solids and fat; it now remains to consider the convenience of the method as compared with the sand and the paper-coil methods which have been extensively used in this country.

(1) Solids and fat are determined in one weighed portion, as in the sand method, while the paper-coil method is usually employed only for the determination of fat.

(2) Asbestos, like sand, does not yield on extraction matters soluble in ether; paper is seldom free from such matters.

(3) The cylinders, like the paper coils, are extracted directly, while in the sand method considerable time and labor is required for transfer.

(4) The ether extract is clear when asbestos or paper are used as absorbents; ether extract from sand must be carefully filtered.

It will be seen that the perforated-cylinder method combines the advantages of both the sand and the paper-coil methods. Some of the other asbestos methods have these same advantages, but for simplicity and convenience the cylinder method can hardly be equaled.

THE DETERMINATION OF FAT IN MILK DRYING PREVIOUS TO EXTRACTION IN AIR AND IN HYDROGEN.

The investigation of this point was proposed by Mr. C. L. Penny, who reports the results of comparative analyses. Although the subject was not brought to my notice until too late to be included with other matters offered for investigation, still a number of results were reported which are of value in this connection.

Mr. Penny reports as follows:

Results obtained from ether extracts of samples of whole milk dried, both in hydrogen and in air.

Sample.	Hydrogen.		Air.	
	Single results.	Mean.	Single results.	Mean.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1st sample	4.09	....	....	....
	4.09	....	....	....
	4.10	....	4.09	....
	4.11	4.09	4.08	4.08
2d sample.....................	3.84	3.84	3.81	3.81
3d sample.....................	3.86	....	3.86	....
	3.87	3.86	3.88	3.87
4th sample.....................	4.46	....	4.43	....
	4.48	4.47	4.48	4.45
5th sample.....................	4.79	....	4.83	....
	4.80	4.79	4.84	4.83
6th sample.....................	....	....	5.19	....
	5.15	5.15	5.15	5.17
7th sample.....................	4.34	....	4.36	....
	4.40	4.37	4.35	4.35
Mean results	....	4.37	....	4.37

NOTE.—The second sample had only one determination in each, and the sixth only one in hydrogen. The samples were all dried in ignited sand, then extracted in anhydrous ether, then pulverized and again extracted. The reagent and methods in the two cases were identical, *i. e.*, when dried in air and in hydrogen. It is my own impression that the results from air drying are the same as those from hydrogen drying.

Messrs. Lyon, Ogden, and Winton, in making their comparative tests, dried with asbestos, using the perforated metal cylinders for drying in air and glass tubes for drying in hydrogen. The average results of the different analyses are given in table V.

TABLE V.—*Comparison of the average results on fat in milk drying previous to extraction in hydrogen and air.*

Analyst.	Number of samples.	Hydrogen.	Air.
		Per cent.	*Per cent.*
C. L. Penny.....	7	4.37	4.37
T. L. Lyon..	1	4.06	4.00
Ogden and Winton..	4	3.52	3.56
All analysts	12	4.06	4.07

These figures indicate that the results by the two methods are practically the same.

THE CALCULATION OF TOTAL SOLIDS IN MILK FROM THE PER CENT OF FAT AND THE SPECIFIC GRAVITY.

The desirability of a formula for the calculation of solids is clearly set forth by Mr. Babcock as follows:[1]

" The introduction of simple and accurate methods for the estimation of fat gives such formulæ a special interest, as by making a direct determination of fat by some of the rapid methods and using the result in a formula for the estimation of total solids an approximately accurate judgment concerning the composition of any milk may be obtained without the use of a chemical balance and in a much shorter time than would be required for obtaining the total solids alone. Moreover, by basing the use of formulæ upon a determination of the fat instead of upon the total solids, the errors arising from inaccurate determinations of the specific gravity fall upon the least valuable constituents of the milk, viz, the solids not fat, so that the chief objections that have been raised to the use of these formulæ no longer exist."

Of the formulæ which may be used for the calculation of the solids only three are here considered.

Let $t=$ per cent total solids, $S=$ specific gravity at 15.5° C. (15° C. in Fleischmann's formula), and $f=$ per cent fat.

Fleischmann:[2] $\quad t = 1.2f + 2.665 \dfrac{100S - 100}{S}$

Hehner and Richmond:[3] $\quad t = \dfrac{f + .2182S}{.859} \quad$ If $\dfrac{S}{t} > 2.5$

Correct the value obtained for t by adding $.05\left(\dfrac{S}{t} - 2.5\right)$

Babcock:[4] $\quad$ Solids not fat $= \left(\dfrac{100S - Sf}{100 - 1.0753Sf} - 1\right) \times (100 - f)2.6$

The calculations may be greatly facilitated by using the tables given in the original papers.

In the case of twenty samples results on solids, fat and specific gravity were reported, thus permitting a comparison of the gravimetric results with those obtained by calculation. Mr. Kilgore determined the specific gravity by the Westphal balance, and Messrs. Ogden and Winton by the pycnometer. The other analysts did not state what method was used. The per cent of fat used in calculating the solids was in each case the average of the results obtained by the different methods. This

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[1] Wis. Ag. Ex. Sta., Rep. 1891, 292.   [3] Analyst, 13, 26
[2] J. Ldw. 1883, 251.   [4] Wis. Exp. Sta. Rep. 1891, 292.

course might have been open to criticism if any but reliable methods had been used, or if the results by the different methods had not agreed closely with each other.

In Table VI are given the averages of the comparative results of the different analysts and of all analysts on samples of whole milk.

TABLE VI.—*Comparison of the average gravimetric with the average calculated results on solids in whole milk.*

Analyst.	Number samples.	Total solids, gravimetric.		Total solids, calculated.		
		Method.	Per cent.	Babcock.	Hehner and Richmond.	Fleischmann.
R. H. Smith	2	Perforated cylinder	13.54	13.82	13.42	13.70
	2	Sand......................	13.58			
B. W. Kilgore..........	3	Perforated cylinder......	13.31	13.82	13.40	13.69
	3	Glass tube, asbestos.....	13.44	14.01	13.59	13.88
	2	Sand......................	13.76	14.21	13.78	14.08
Charles Ryan..........	1	Perforated cylinder......	13.67	13.80	13.42	....
	1	Sand......................	13.99			
	1	Paper coil................	10.70			
	1	Dried alone..............	13.55			
F. S. Shiver	3	Perforated cylinder......	11.43	11.74	11.41	11.68
	5	In dish, asbestos	12.21	12.44	12.09	12.38
	3	Sand......................	12.27	12.50	12.14	12.43
	2	Dried alone	12.21	12.35	12.01	12.30
A. W. Ogden and A. L. Winton.	4	Perforated cylinder......	12.88	13.03	12.67	12.90
	4	Sand......................	12.76			
	4	Dried in hydrogen.......	12.73			
	2	Dried alone..............	13.52	13.68	13.27	13.56
All analysts	12	Perforated cylinder......	**12.74**	13.04	**12.66**	12.92
	8	Other asbestos methods..	**12.67**	13.03	**12.65**	12.94
	11	Sand......................	**12.96**	13.24	**12.86**	13.13
	4	Dried alone..............	**12.86**	13.01	12.64	**12.93**
	4	Dried in hydrogen	**12.73**	13.03	**12.67**	12.90

The averages of all the results on total solids by each of the five gravimetric methods are in all cases lower than those calculated by Babcock's or Fleischmann's formula, and in the case of four of the five methods slightly higher than by the formula of Hehner and Richmond. In Table V the averages of the calculated results of all the analysts agreeing closest with those by direct analysis are printed in heavy-faced type. It will be seen that the closest agreement was obtained by the formula of Hehner and Richmond when solids were determined by drying with sand or asbestos in air or with asbestos in hydrogen, but by Fleischmann's formula when the milk was dried alone for determination of solids.

In the case of three analyses of skim milk (see Table I), two by Mr. Kilgore and one by Mr. Shiver, the calculated solids by Babcock's formula were closest to the gravimetric results.

On a single sample of cream the calculated result by the formula of Hehner and Richmond agreed within 0.10 per cent with that by direct analysis.

Although in the analyses of whole milk here reported the formula of Hehner and Richmond gave the most satisfactory results, still further trials are needed before arriving at a final conclusion. I would suggest that the investigation be continued during the ensuing year, taking every possible precaution in the analytical work, particularly in the determination of the specific gravity.

Mr. WINTON. I make but one recommendation, which is that for the determination of total solids we adopt the Babcock asbestos method,

which is used in at least half a dozen of our stations. It is employed in Wisconsin, at both the New York stations, in Illinois, and, I think, in Indiana. We have now but two official methods. One is the Adams paper-coil method, which is given first place, and the other, called the alternate method, devised by Mr. Babcock, except that it directs drying in a tube in a current of air. I understand that Mr. Babcock himself has discarded that method, and now uses what I call the cylinder method. I doubt whether the tubes are now used at all. I think it would be well to substitute the proposed method for the other, and I would also suggest that it be given the first place, as I think it a very much better method than the paper-coil method. I have prepared a brief description of the proposed substitute for the alternate method, and will read it.

Mr. FREAR had used the asbestos method, and in nearly every case where comparisons had been made between the direct and indirect determinations of fat he had found the results practically identical.

Mr. WOLL said that the asbestos method had been employed in the Wisconsin station during the past four years. They had gained a good deal of experience in handling and preparing the asbestos. After ignition the asbestos was treated with hydrochloric acid solution and washed with distilled water until the fluid was no longer acid. It was then dried in an air bath and kept in stoppered bottles. When determinations were made the tubes were placed in a drying oven previous to weighing them—first in a drying oven and then in a desiccator. Under those conditions very good results were obtained. They used perforated copper tubes coated with a thin layer of platinum, which would wear as long as any others.

The PRESIDENT stated that when a poor or even a good quality of asbestos was used it was unnecessary to treat with acid. The so-called asbestos was not asbestos at all, and would gelatinize when treated with even a moderately strong acid. He saw no necessity for including such precautions in the description of the method.

Mr. TELLER asked for what length of time the reporter proposed to extract the tubes.

Mr. WINTON replied that he had found two hours a sufficient time, but would recommend a longer extraction.

On motion of Mr. Van Slyke, the recommendation was adopted.

Mr. VAN SLYKE read the following papers:

THE DETERMINATION OF CASEIN IN COWS' MILK.

By L. L. VAN SLYKE.

The methods originally proposed by Hoppe–Seyler and Ritthausen have been commonly employed for the separation and determination of casein in cows' milk. In both methods the milk is diluted with water and a small amount of acetic acid is added. The precipitation is rendered complete in one case by raising the temperature to 40° C., and in the other by passing carbon dioxid through the mixture

at ordinary temperatures. The precipitate is filtered, washed, first with water and then with ether, to remove fat, and is finally dried and weighed on filter.

The absence of specific directions touching several steps of the operation led me to investigate some of the conditions of these methods, among which were the following:

(1) Comparison of the two methods.

(2 Influence of amount of acid used in precipitating casein.

(3) Use of different acids in precipitating casein.

(4) Influence of temperature and time of digestion on precipitation of casein.

(5) The precipitation of casein in fresh and in old milk.

(6) The use of preservatives in keeping milk and influence on determination of casein.

The tedious method involved in washing the precipitated casein free from fat by ether and in subsequent drying and weighing on filter was entirely discarded. The precipitated casein was washed by decantation and on filter two or three times. The filter and contents were then treated by the ordinary Kjeldahl method for the determination of nitrogen, the official factor 6.25 being used to convert the amount of nitrogen into an equivalent of casein, when this was desired. This use of the Kjeldahl method in determining the amount of casein was suggested and employed some time since by several chemists, and has recently been very generally used in connection with dairy products. Its advantages over the old method in point of accuracy and simplicity are too evident and too well known to deserve further mention. Much credit is due to Mr. A. L. Knisely, who did valuable work in carrying out the analytical details of the investigation.

COMPARISON OF RESULTS OBTAINED BY PRECIPITATING CASEIN IN MILK AT A TEMPERATURE OF 40° C. WITHOUT CARBON DIOXID AND WITH CARBON DIOXID AT ORDINARY TEMPERATURES.

The method employed in doing the work was briefly as follows: About 10 grams of milk were used in each case. This amount of milk was diluted with water at 40° to 42° C. to 100 cc. and then 1 or 2 cc. of a solution containing 10 per cent of acetic acid were added and the solution was carefully stirred with a glass rod. The resulting precipitate was washed two or three times by decantation and on filter, and finally the filter and contents were digested according to the Kjeldahl method for determining nitrogen. In the other case the milk was diluted to 100 cc. with cold water, the same amount of acetic acid was used as before, and then a stream of carbon dioxid was passed through the solution until the liquid above the precipitate was clear, or very nearly so. The subsequent filtration, washing, and determination of nitrogen were made as above.

The following results were obtained by the two methods with two different samples of milk, triplicate determinations being made in each case:

Method A.	Method B.
Precipitation at 40° C.	*Precipitation with aid of carbon dioxid.*
Sample No. 1:	Sample No. 1:
(a) .... 0. 475 per cent nitrogen.	(a)... ...... 0. 486 per cent nitrogen.
(b) .... 0. 474 per cent nitrogen.	(b).......... 0. 480 per cent nitrogen.
(c) .... 0. 469 per cent nitrogen.	(c).......... 0. 461 per cent nitrogen.
Average.. 0. 473 per cent nitrogen.	Average .. 0, 476 per cent nitrogen.
Sample No. 2:	Sample No. 2:
(a) .... 0. 458 per cent nitrogen.	(a) .... 0. 454 per cent nitrogen.
(b) .... 0. 458 per cent nitrogen.	(b) .... 0. 444 per cent nitrogen.
(c) .... 0. 449 per cent nitrogen.	(c).......... 0. 455 per cent nitrogen.
Average.. 0. 455 per cent nitrogen.	Average.. 0. 451 per cent nitrogen.

The results obtained indicate—

(1) That the same results practically are obtained whether we precipitate the casein in cows' milk at a temperature of 40° C. without carbon dioxid or at ordinary temperatures with the aid of carbon dioxid. In sample No. 1 the results differed by 0.003 per cent of nitrogen; in sample No. 2, by 0.004 per cent of nitrogen, the excess being first with one method and then with the other.

(2) That the individual results obtained by several determinations of the same milk agree closely by either method, somewhat closer concordance being given by precipitation at 40° C. Thus, in sample No. 1 the greatest difference in three determinations was 0.006 per cent of nitrogen at 40° C. and 0.025 per cent with the other method. In sample No. 2 the greatest difference was 0.009 per cent of nitrogen at 40° C. in three determinations, and 0.011 per cent with the other method.

INFLUENCE OF AMOUNT OF ACID IN PRECIPITATING CASEIN.

The acid used was a solution containing 10 per cent of acetic acid. This dilute acetic acid was used in amounts varying from 0.5 to 5 cc. To about 10 grams of milk sufficient water at 40° C. was added to dilute to 100 cc., and the acetic acid was then added. The operation was completed as already described above.

Below the results are given in tabulated form:

Sample.	Amount of acid used.	Character of filtrate.	Nitrogen found in precipitate.
	cc.		*Per cent.*
No. 1......	0.5	Slightly cloudy...	0.458
	1.0	Clear	0.458
	2.0	......do	0.449
	3.0	......do	0.441
No. 2......	1.0	......do	0.475
	1.5	......do	0.474
	2.0	......do	0.469
	3.0	Slightly cloudy...	0.450
No. 3......	0.5	Milky	0.117
	1.0	Quite cloudy	0.471
	1.5	Clear	0.545
	2.0	......do	0.550
	2.5	......do	0.545
	3.0	......do	0.537
	4.0	Slightly cloudy...	0.527
	5.0	Milky.............	0.340
No. 4......	0.5	......do	0.059
	1.0	Cloudy	0.551
	1.5	Clear	0.550

The results obtained show—

(1) That in one sample 0.5 cc. of acid gave a maximum amount of nitrogen in the precipitate, while in two other samples the amount of nitrogen obtained in the precipitate was very low. In each instance the filtrate was cloudy or very milky in appearance.

(2) That in three samples out of four the use of 1 cc. of acid gave highest results, the filtrate being clear or only perceptibly cloudy.

(3) That in most instances the use of 1.5 cc. of acid gave highest results, with clear filtrate in every case.

(4) That the use of 2 cc. of acid gave very nearly the same results as the use of 1.5 cc. of acid in most cases, the filtrate being clear.

(5) That the use of 2.5 cc. or more of acid gave lower results, the decrease becoming greater with increase of acid and the character of the filtrate becoming more t 'bid.

(6) That with fresh milk the best results can be secured in determining casein by using from 1 to 2 cc. or about 1.5 cc. of a 10-per-cent solution of acetic acid.

(7) The completeness of precipitation is quite fairly indicated by the character of the filtrate. If the precipitation is incomplete as a result of too little acid precipitant, the filtrate will be more or less turbid, varying from opaque milkiness to slight cloudiness. If, on the other hand, an excess of acid is used, thereby causing more or less of the casein to remain in solution, the filtrate will be more or less turbid, according to the amount of casein in solution. In some cases a filter paper may allow some precipitated casein to pass through and render the filtrate turbid. In such cases two or three successive filtrations generally serve to remove the casein and give a clear filtrate.

THE USE OF DIFFERENT ACIDS IN PRECIPITATING CASEIN.

Can some of our common acids be used more effectively to precipitate casein than acetic acid? A comparison was made between acetic acid and each of the three following acids: (1) Lactic, (2) sulphuric, and (3) hydrochloric. In the comparisons the milk was treated as above described, using 1.5 cc. of a 10-per-cent solution of acetic acid. A sample of the same milk was also treated with varying amounts of the other acids, the operation being completed as before described. In every case a 10-per-cent solution of acid was used.

The results are tabulated below:

Kind of acid used.	Amount of acid used.	Character of filtrate.	Character of precipitate.	Nitrogen in precipitate.
	cc.			Per cent.
Acetic acid	1.5	Clear	Flocculent	0.395
Lactic acid	.5	Milky	......do	.074
Do	.75	Slightly cloudy	......do	.396
Do	1.0	Clear	......do	.394
Do	1.5	Milky	......do	.268
Acetic acid	1.5	Clear	Flocculent	.465
Sulphuric acid	.5	....do	Somewhat gelatinous	.470
Do	.75	....do	......do	.457
Do	1.0	Cloudy	......do	.417
Acetic acid	1.5	Clear	Flocculent	.464
Hydrochloric acid	.3	Cloudy	Gelatinous	.410
Do	.5	....do	......do	.404
Do	.75	Milky	......do	.199

The results secured indicate:

(1) That lactic acid gives results closely agreeing with those obtained by the use of acetic acid when about 1 cc. of a 10 per cent solution of lactic acid is used, but if 0.5 cc. more or less than 1 cc. of lactic acid are used the results are very low. The precipitate was flocculent and filtered readily.

(2) That a 10 per cent solution of sulphuric acid when used to the extent of 0.5 cc. gave results closely agreeing with those given by use of acetic acid, but that when more than 0.5 cc. was used the results became lower. The filtrate was clear, but the casein did not separate in flocculent form, having a tendency to become gelatinous. Several filtrations were required to get a clear filtrate, and filtration was slow.

(3.) That very unsatisfactory results were given by the use of hydrochloric acid, whatever amount was used. The filtrate was cloudy or milky, the precipitate gelatinous, the filtration very slow, and the results low.

(4.) The use of acetic acid gave much more satisfactory results, all things considered, than the use of any of the other acids that were tried.

INFLUENCE OF TEMPERATURE AND TIME OF DIGESTION ON PRECIPITATION OF CASEIN.

In making these experiments the same amount of acetic acid was used and all other conditions were kept uniform, except those pertaining to time and temperature of digestion after precipitation.

Temperature of digestion.	Time of digestion.	Character of filtrate.	Nitrogen in precipitate.
° C.	*Minutes.*		*Per cent.*
30	5	Clear	0.448
40	0	....do	.475
40	1	....do	.479
40	2	....do	.481
40	2 to 3	....do	.471
40	10	....do	.477
40	20	....do	.484
45	0	....do	.468
45	2	....do	.476
45	5	....do	.483

The following results were obtained:

(1) When the precipitate was digested at 30° C. for five minutes, the results were lowest.

(2) When the precipitate was digested at 40° C., without standing or with standing one, two, three, ten, or twenty minutes, the results were practically uniform. The greatest difference obtained did not exceed 0.01 per cent nitrogen.

(3) When the precipitate was digested at 45° C. for periods of time varying from a few seconds to five minutes, the results were fairly uniform, the greatest difference being 0.015 per cent of nitrogen. As compared with digestion at 40° C., digestion at 45° C. gave essentially the same results.

(4) It would therefore seem that variation of temperature 2° or 3° from 40° C. and variation of time of digestion after addition of acetic acid exercise comparatively little influence upon the results. However, in practice we have felt it desirable to adhere pretty closely to uniformity of temperature.

THE PRECIPITATION OF CASEIN IN FRESH AND IN OLD MILK.

The fact is well known that milk undergoes rapid and complete changes when left to itself at ordinary temperatures. In the course of our work the thought suggested itself that as a result of these changes the casein might be so changed that the method used in determining the casein of fresh milk might not be applicable to milk that had undergone more or less change, or that the formation of lactic acid might make necessary the use of less acetic acid than is used in precipitating the casein of fresh milk. It was generally found that after milk had stood at a temperature of 17° to 22° C. for twenty-four hours or more, the use of 1.5 cc. of a 10 per cent solution of acetic acid gave low results with turbid filtrate. In some cases the casein was almost entirely redissolved and passed into the filtrate. The only method of handling milk under such circumstances was to add the acetic-acid solution to the milk diluted with water at 40° to 42° C. in small portions of a few drops at a time, stirring after each addition and continuing the addition until the liquid above the precipitate became clear or nearly so. But even with this treatment there was more or less loss. The terms "fresh" and "old" milk are necessarily rather vague. Much depends upon the conditions under which milk is kept. The extent

of change, rather than the actual age, should form the basis of distinction. Coagulated milk always gave lower results than did the same milk when fresh. The author has in progress an investigation relating to the detailed changes that take place in the nitrogen compounds of milk on standing under ordinary conditions.

Sample.	Age of milk.	Nitrogen in precipitate.	Amount of loss.
		Per cent.	
No. 1..............	Fresh.....	0.429	....
	7 days	.378	0.051
No. 2..............	Fresh	.447	....
.	1 day	.427	.020
	3 days	.428	.019
	7 days	.428	.019
No. 3..............	Fresh.....	.428	....
	1 day	.410	.018
No. 4..............	Fresh.....	.438	....
	1 day	.415	.023
No. 5..............	Fresh.....	.624	....
	1 day	.617	.007
No. 6..............	Fresh.....	.397	....
	1 day	.397	....
No. 7..............	Fresh.....	.466	....
	1 day	.445	.021

The results are as follows:

(1) When milk was kept at a temperature of 17° to 22° C., for twenty-four hours the amount of nitrogen in casein was found, in most cases, to be from 0.007 to 0.020 per cent less than in fresh milk.

(2) In case of one milk kept seven days the amount of nitrogen as casein was 0.051 per cent less than in fresh milk, while, in another case, it was 0.019 per cent less.

(3) In general it was found necessary, in order to get results at all satisfactory, to use less than 1.5 cc. dilute acetic acid to precipitate casein in milk that was not quite fresh.

(4) While good results may, in exceptional cases, be secured in determining casein in milk that is twenty-four hours or more old, without any modification of the method given above for determination of casein, the accuracy of such determinations must be regarded as uncertain.

THE INFLUENCE OF THE USE OF PRESERVATIVES ON THE DETERMINATION OF CASEIN IN MILK.

When it is necessary to determine the casein in samples of milk which are 1 or 2 days old the question arises, in view of what has just gone before, "Is it possible to determine casein accurately in milk that is not fresh?" In order to test the question, finely pulverized mercuric chlorid was used in the proportion of about 1 part of mercuric chlorid to 2,000 parts of milk, by weight. A sample of milk was analyzed fresh, and portions were set aside for varying periods of time, some with and some without the addition of mercuric chlorid. Determinations of casein were made from time to time, using less than 1.5 cc. of acetic acid. The results were as follows:

Age of milk.	Nitrogen in precipitate when no mercuric chlorid was used.	Amount of loss.	Nitrogen in precipitate when mercuric chlorid was used.	Amount of loss.
	Per cent.		Per cent.	
Fresh..............	0.447	....	0.447	....
1 day	0.427	0.20	0.444	0.003
3 days	0.428	0.19	0.444	0.003

The foregoing results appear to indicate—

(1) That the use of mercuric chlorid in fresh milk largely prevents or retards the occurrence of those changes that affect the determination of casein.

(2) That even when mercuric chlorid is used in milk it is generally desirable to use less than 1.5 cc. of acetic acid.

(3) That the use of mercuric chlorid in the proportions stated did not precipitate in appreciable quantity any nitrogen compounds, such as albumen.

GENERAL SUMMARY.

We may summarize the results of our work as follows:

(1) Precipitation of casein in cows' milk gives essentially the same results whether made at 40° C. or at ordinary temperatures with the aid of carbon dioxid, other conditions being uniform.

(2) Between 1 and 2 cc. of a 10 per cent solution of acetic acid, generally about 1.5 cc., gave the best results.

(3) The use of lactic acid, sulphuric acid, and hydrochloric acid did not give as satisfactory results as did the use of acetic acid.

(4) A variation of a few degrees from 40° C. and a variation of time of digestion after the addition of acetic acid exercised comparatively little influence on results.

(5) The precipitation of casein in milk that had undergone noticeable change was generally found to give lower results than in the case of the same milk when fresh, even when less than 1.5 cc. of acetic acid were used. Coagulated milk always gave low results compared with fresh milk.

(6) The use of mercuric chlorid in fresh milk, in the proportion of 1 part of the former to 2,000 parts of milk, largely prevented or retarded the occurrence of those changes that affect the determination of casein in old milk.

(7) In a general way, the character of the filtrate serves as a fairly reliable guide in regard to the completeness of the precipitation of casein from milk. When the filtrate is perfectly clear or only perceptibly cloudy the precipitation is generally complete; but when the filtrate is decidedly turbid or milky and two or three repeated filtrations do not serve to remove the turbidity the precipitation will generally be found incomplete, and such a determination, if carried to the end, will give low results.

DETAILED DESCRIPTION OF METHOD USED IN DETERMINING THE CASEIN OF COWS' MILK.

(1) In fresh milk.

Weigh out about 10 grams of milk, dilute in a beaker with about 90 cc. of water at 40°–42° C., and add at once 1.5 cc. of a solution containing 10 per cent of acetic acid, by weight. Stir with a glass rod and let stand three to five minutes or longer. Then decant on filter, wash two or three times with cold water by decantation, and then transfer precipitate completely to filter. Wash once or twice on filter. The filtrate should be perfectly clear or very nearly so. If the filtrate is not clear when

it first runs through, it can generally be made so by two or three repeated filtrations, after which the washing of the precipitate can be completed. The washed precipitate and filter paper are then digested as in the regular Kjeldahl method for the determination of nitrogen, and this determination of nitrogen is completed as usual. To calculate the nitrogen into an equivalent amount of casein, multiply the amount of nitrogen found by the factor 6.25. Ordinarily for this purpose milk can be regarded as "fresh" when it does not show marked development of lactic acid.

(2) In old milk.

When milk has undergone such change as to show marked development of lactic acid the method above given can not generally be relied upon to give accurate results in determining casein. So far as our results go, they indicate that we can not with positive accuracy determine casein in such milk by any method now known, and results obtained with changed milk must be regarded as only approximate. If, however, 1 part of finely-powdered mercuric chlorid be added to 2,000 parts of milk when fresh, the changes which would otherwise take place are prevented or retarded, so that milk treated in this manner may be used some days after standing for the determination of its casein. In such cases the method given above for fresh milk may be followed, except that the acetic acid should be added in small portions, a few drops at a time, stirring after each addition, and continuing the addition of acetic acid until the liquid above the precipitate becomes clear or very nearly so.

THE DETERMINATION OF FAT IN CHEESE.

By L. L. Van Slyke.

At the New York State Agricultural Experiment Station (Geneva) we have used for two years or more a modification of the copper-sulphate method for determining fat in butter with satisfactory results; and we have also made some experiments with a view to simplifying the official method now used for the determination of fat in cheese. The method employed is this, briefly: From the prepared sample of cheese about 2 grams are weighed out and placed in a cone of copper foil made of a size suitable to slide into the test tube containing copper sulphate. The test tube used to place in the extraction tube has a perforation in the bottom. In packing this tube asbestos or extracted cotton or glass wool is placed in the bottom. Upon this is placed a mixture composed of equal parts of anhydrous copper sulphate and pure incinerated sand, filling the tube for about 2 inches. Above this is placed a little asbestos or other suitable material, and upon this is placed the copper-foil cone containing the cheese to be extracted. The lower end of the test tube is wrapped about with filter paper to prevent particles of asbestos or copper sulphate being carried down through the perforation into the extraction flask. The extraction is carried on as usual. After extracting five hours the copper-foil cone is removed and the partially-extracted cheese is ground with pure sand in a mortar. This partially-extracted cheese is brittle and grinds up about as easily as a dry cracker. After grinding with the sand the cheese is replaced in the cone and the extraction continued for ten hours. As to manipulation, this method can be carried out rapidly; in the grinding there is no danger of loss of fat, because the cheese is largely extracted in five hours and little fat remains in the residue that is ground.

Below are some results secured by the official method, by the suggested modified method, and by extraction after drying in the official way. One cheese was an Edam five months old and the other two were green cheeses, which are least easy to extract.

Kind of cheese.	Age.	Official method.	Proposed modification.	Extraction after drying 10 hours.
		Per cent.	Per cent.	Per cent.
Edam cheese................	5 months	(a) 29.99	(a) 32.18	(a) 32.57
		(b) 33.88	(b) 32.24	(b) 32.54
		(c) 32.04	....	(c) 32.47
Average..............	....	31.97	32.21	32.53
Factory cheddar............	Green.........	(a) 38.94	(a) 35.63	(a) 36.49
		(b) 36.57	(b) 36.59	(b) 36.21
		(c) 34.99	(c) 36.59	(c) 35.80
Average	....	36.83	36.27	36.17
Factory cheese	Green.........	(a) 36.00	(a) 36.35	(a) 36.51
		(b) 35.33	(b) 36.73	(b) 35.83
		(c) 36.10	(c) 36.41	(c) 38.27
Average...............	....	35.81	36.50	36.87

The results, briefly summarized, show that by the modification employed the results obtained in determining the fat in different samples of the same cheese agreed more closely than by either of the other methods.

THE DETERMINATION OF ACIDITY IN MILK.

By L. L. Van Slyke.

It is frequently desirable to determine the amount of acid in milk. The usual method has been to use phenolphthalein as an indicator, and to titrate against a standardized solution of caustic soda, calculating the result as lactic acid. The question occurred to me as to how much the color of the milk obscured the delicacy of the end-reaction, and whether the method did not give too high results.

In order to study the question, a sample of milk was examined when ten, twenty-four, and forty-eight hours old. In one portion of 10 grams the milk was titrated against a standardized solution of caustic soda, with phenol-phthalein as indicator; in a second portion the milk was diluted with water to about ten times its volume, and then similarly titrated. In a third portion, the casein was precipitated with a known quantity of acetic acid and the filtrate titrated as before, the known amount of acid added being deducted from the total amount of acid found.

The results are tabulated below:

Age of milk.	Lactic acid in milk.		
	Direct titration without dilution.	Titration after dilution.	Titration after precipitating casein with acetic acid.
	Per cent.	Per cent.	Per cent.
Ten hours	.2097	.1458	—0.3024
Twenty-four hours	.7299	.6408	.2394
Forty-eight hours	.8946	.8343	.4581

The following is a statement of results:

(1) Direct titration without dilution of milk gave from 0.064 to 0.090 per cent more of lactic acid than did titration after dilution; the average difference was 0.071 per cent. This difference was equivalent to from 7.25 to 43.8 per cent of the lactic acid present, with an average of 13.2 per cent.

(2) With the sample of fresh milk, an apparently curious result was obtained by precipitating the casein with a known quantity of acetic acid and determining the acid in the filtrate. The amount of acetic acid added to precipitate the casein was equivalent to 1.4706 per cent of lactic acid, and, at first thought, we should expect to get this amount in the filtrate plus whatever amount of lactic acid there might be in the milk; but, instead of getting something like 1.62 per cent, as we expected, we actually found by titration only 1.17 per cent; that is, after adding an equivalent of 1.47 per cent of lactic acid, we recovered about 0.3 per cent less than we added, thus giving a negative result. The only explanation of this behavior is that the acetic acid used in precipitating the casein was in part retained by the casein and was not recoverable in the filtrate.

After the same milk had stood twenty-four and forty-eight hours, some acid was found in excess of that added, the excess being 0.24 to 0.46 per cent and averaging 0.35 per cent. The amount of acid found in this way was less than one-half that found by either of the other methods.

Another sample of milk was taken and acid determinations made in various portions as before, except that, in place of acetic acid, 10 cc. of a saturated solution of magnesium sulphate were used to precipitate the casein in 10 grams of milk, the mixture being heated to 40°.6, and the filtrate being used for titration as before. The results are tabulated below:

Age of milk.	Lactic acid in milk.		
	Direct titration without dilution.	Titration after dilution.	Titration after precipitating casein with magnesium sulphate.
	Per cent.	Per cent.	Per cent.
Ten hours..................	.1872	.1143	.1665
Twenty-four hours.........	.8550	.8028	.3753
Forty-eight hours..........	1.0008	.9186	.4743

The results are as follows:

(1) Direct titration without dilution gave higher results than did titration after dilution, the amount of excess varying from 0.0522 to 0.0629 per cent of lactic acid, and averaging 0.0558 per cent. This difference was equivalent to from 5.50 to 55 per cent of the lactic acid present, with an average of about 10 per cent.

(2) The results given by precipitating casein with magnesium sulphate and determining the acid in the filtrate were lower than by either of the other methods, except in one case, being, on an average, 0.3423 per cent lower than by titration without dilution and 0.2832 per cent lower than by titration after dilution. The results secured by removing casein with magnesium sulphate were 54.4 per cent of those secured by titration after dilution, being somewhat higher than those secured when acetic acid was used as precipitant and the lactic acid determined indirectly.

SUMMARY.

Our results, so far as they go, show that the determination of acid in milk by direct titration without dilution gives results that are considerably too high, while

results obtained by dilution are probably higher than the true results warrant. While I am not prepared yet to say that removal of casein by use of magnesium sulphate gives absolutely correct results, I believe it to be more reliable than the method now commonly used.

The President stated that Mr. Winton had a paper to present on milk analysis. It was as follows:

THE BABCOCK GRAVIMETRIC METHOD FOR THE DETERMINATION OF SOLIDS AND FAT IN MILK—(DRYING WITH ASBESTOS IN A PERFORATED METAL CYLINDER).

By A. L. WINTON.

Asbestos has been adopted as the absorbent in milk analysis, at a number of the experiment stations in the United States, owing to the advantages which it possesses over paper, sand and other material. The asbestos best suited for the purpose is a woolly variety free from fine or brittle material, which should be ignited previous to use in order to remove hygroscopic moisture and burn off any organic matter.

Of the various dishes, tubes, etc., which have been used for holding the asbestos during the drying and extracting, the perforated metal cylinder devised by Dr. S. M. Babcock has proved in my experience the most satisfactory. It consists of a tube of perforated sheet metal, about 60^{mm} long and 20^{mm} in diameter, closed 5^{mm} from one end by a disk of the same material. The perforation should be 0.7^{mm} in diameter and about 0.7^{mm} apart. Sheets of perforated tin plate or brass are obtainable from which a tinsmith can easily make the cylinders.

The method for the determination of solids and fat is as follows: The cylinder is filled loosely with 1.5 to 2.5 grams of freshly ignited asbestos, cooled in a desiccator and weighed. A weighed quantity (about 5 grams) of milk is then introduced and the whole dried at 100° to constant weight which usually requires about 4 hours. During the first part of the drying provision should be made for the escape of moisture from the bath.

The cylinder with its contents is next placed in an extractor and the fat extracted with anhydrous ether. The fat may be determined by direct weighing of the fat in the usual manner or by difference, drying the extracted cylinders at 100° C.

During the past year a comparison of this method with other standard methods was undertaken with the coöperation of the following chemists of the Association of Official Agricultural Chemists: B. W. Kilgore, Raleigh, N. C.; R. H. Smith, Amherst, Mass.; Chas. Ryan, Fort Collins, Colo.; F. S. Shiver, Fort Hill, S. C.; T. L. Lyon, Lincoln, Nebr.; R. E. Blouin, Baton Rouge, La., and A. W. Ogden, New Haven, Conn.

Determinations of solids and fat were made on 21 samples of whole milk and on a number of samples of skim milk, cream and buttermilk. The results of these tests (which will be published in detail in the proceedings of the association) prove the accuracy of the Babcock method. Considering only the average results on whole milk, the Babcock method gave 0.04 per cent more fat and 0.04 per cent less solids than the sand method, 0.03 per cent more fat than the Adams paper coil method and 0.02 per cent more solids than drying the milk alone in a dish. The results agreed also with those by other standard methods.

With regard to convenience it may be said that the method combines all the advantages of the sand and the paper coil methods, viz: solids and fat are determined in one weighed portion, the cylinders are used for both drying and extracting without transfer, the asbestos does not contain matters soluble in ether for which correction must be introduced and finally the extract does not require filtering.

Very good results on fat can also be obtained by difference drying the extracted residue, thus furnishing a valuable check on the results by direct weighing and a resource in case of accident to the extract.

No further papers being presented, Mr. Atwater addressed the association on the subject of international coöperation in methods of analysis.

Mr. ATWATER. A number of you will recall that in the congress of chemists, at the meeting held yesterday, a motion was passed looking toward the securing of some international coöperation in the study of methods of investigation. As I remarked there, I do not believe that it is going to be possible to bring about anything of that sort and make it successful immediately. I do believe, however, most firmly that the time is getting ripe for it. I have had the opportunity lately of talking with a very considerable number of our colleagues and fellow workers in several of the different countries of Europe, and have been pleased to notice the fact that they seemed to be thinking, as some of us are, that the time was coming when we should be working together a little more. At the meeting of the association of experiment stations in the German Empire, which corresponds in a measure to our association of colleges and stations, and perhaps more to this association, the matter was brought up, and the discussion upon it, though brief, was quite favorable, and a motion was passed, though it was done hurriedly, being near the close of the convention, that the matter be referred to the officers of that association. That looked toward the possibility of something being done. It seems to me it could certainly do no harm, and might be useful, if this association would put itself on record as favorable to such coöperation, and would likewise take some steps toward an actual realization of it. Such a step might be found, provided that should meet with the approval of the association, in authorizing or instructing the executive committee to take such action as might seem to it desirable. It has occurred to me that perhaps there might be some coöperation between this association and the Office of Experiment Stations of the Department of Agriculture, which office is in such direct relations with many of our European investigators as to be able, perhaps, to materially facilitate at least the preliminary negotiations which should look toward such coöperation. I hesitate to make any very definite proposal, but will simply make this suggestion, assuring you that if it does not meet with your favor I shall not take it illy. The suggestion I would make, then, is that, in the first place, this association express itself as favorable to such coöperation, and as desirous that it may be brought about; and, second, that the executive committee of this association be instructed or authorized—whatever word will be best—to take such steps as may seem to it desirable in this direction. If it shall seem best to the association to adopt the suggestion to coöperate with the office of Experiment Stations in Washington, I think that might not be out of the way, but would rather know a little more how the matter would strike the association before making any definite suggestion.

Mr. HUSTON. Perhaps the best evidence of our willingness to coöperate with Europeans in matters relating to the analysis of products

coming within our sphere can be found in the fact that in the case of potash our reporters receive more data from the other side of the water than from our own members. There can be no doubt of the good will of this association in the matter referred to.

Mr. ATWATER. Of course you are not unaware of this—my impression may be a wrong one; it is, however, definite—that a fuller and more successful coöperation could be obtained if the gentlemen over there saw not only that our association, through its reporters, was taking hold of the practical work, but if they were to see that we are so much in earnest about it that the association itself officially desires such coöperation; and you will observe that the suggestion which I made was so guarded as not to interfere with, but rather to emphasize and further, the most excellent work which has already been begun.

Mr. HUSTON. I think it would be better to leave the matter in the hands of the reporters rather than with the executive committee.

Mr. FREAR. It seems to me that the reporters do not constitute a representative body which can readily be united for action in any coöperative attempt made, for instance, by an appointed committee of the association. Therefore I would move that the executive committee of this association be authorized to coöperate with any other committee in any way that seems to best further the desired end.

Mr. VAN SLYKE agreed that the association ought to promptly place itself on record as recognizing the desirability of coöperation. He seconded Mr. Frear's motion.

Mr. ATWATER. If the association should feel inclined to, in the first place, express itself definitely, and, in the next place, authorize or direct its executive committee to take such steps as might seem advisable, it would leave them a little more freedom to act.

Mr. FREAR.–I believe that the passage of a resolution of this general character, without further action, will unmistakeably place us on record as favoring international coöperation. I have no objection to the use of the phrases suggested by Mr. Atwater. I do not care what phrase is used to arrive at the result.

Mr. ATWATER. Would Mr. Frear, then, accept an amendment of his phraseology something like this :

Resolved, That in the judgment of this association a more general international coöperation in the study of methods of analysis is desirable.

Resolved, That the executive committee of the association be authorized to take such steps as they may find expedient to secure this end.

Mr. FREAR accepted the amendment and the resolutions were adopted. The president called for the report on cattle foods.

Mr. WOLL presented the following paper:

REPORT ON ANALYSIS OF CATTLE FOODS.

By F. W. WOLL and GEO. L. TELLER.

The reporter on foods and feeding stuffs low in fiber on January 13 sent a circular letter to experiment station chemists likely to be interested in the analysis of cattle foods, asking their coöperation in an investigation of methods of fodder analysis for the coming year. Twenty-seven chemists signified their desire to take part in the work, and on February 6, samples of corn meal and of linseed-oil meal were accordingly forwarded to each one, with an outline of the particular points to be studied. Believing that more valuable data would be obtained by concentration rather than doing a little all along the line, only two methods were taken up, viz, ether extract and crude fiber. Under the former the purification by animal charcoal and the extraction without previous drying of the fodder were included, and under the latter Hönig's method for determination of crude fiber and starch.

Of the chemists intending to take part in the investigation the greater number found it impossible to do so, in the majority of cases owing to the great amount of extra work laid upon each one during the year, directly or indirectly, through the World's Columbian Exposition. Your reporters especially regret that they could find time to do only a small part of the analytical work planned. It is believed, however, that what has been done by the various analysts will prove of considerable value and throw additional light on the problems studied.

MOISTURE.

The circular letter asked for a determination of moisture in the samples sent out by the official method, and several chemists extended the determinations to drying in the air. The following results were reported:

Determinations of moisture in corn meal and oil meal.

Analyst.	Dried in hydrogen.		Dried in air.	
	Sample 1, corn meal.	Sample 2, linseed meal.	Sample 1, corn meal.	Sample 2, linseed meal.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1. Patterson, Mississippi	10.27	8.26	8.97	6.73
2. Shiver, South Carolina	10.20	8.42	9.30	7.19
3. Kilgore, North Carolina [1]	8.99	7.35	....	....
4. Rossman & Harrison, Michigan	10.52	8.47	....	....
5. Voorhees, New Jersey	10.18	7.99	....	....
6. Bartlett, Maine	10.25	8.69	10.21	8.57
7. Huston, Indiana [2]	9.74	8.15	....	....
8. Teller, Arkansas	10.30	8.85	....	....
9. Runyan, U. S. Department of Agriculture	9.55	8.08	....	....
10. Woll, Wisconsin [3]	9.55	7.58	....	....
Average	9.96	8.18	....	....
Greatest variation from average	.97	.83	....	....
Greatest variation from average in per cent of average	9.7	10.1	....	....
Difference between highest and lowest	1.53	1.50	....	....
Difference between highest and lowest in per cent.	15.4	18.3	....	....
Average of 1, 2, and 6	10.24	8.46	9.49	7.50

[1] Six hours at 100° C.   [2] Four hours.   [3] Ten hours.

. The difference between the highest and the lowest results found by the official method is, for corn meal 1.53 per cent, or 15.4 per cent of the moisture present; and for oil meal 1.50 per cent, or 18.3 per cent of the moisture present. The results, therefore, come outside of the 15 per cent limit adopted by Prof. Caldwell a few years ago in our association work; even if such was not the case, if a considerably better agreement was not obtained, the results could hardly, as it seems to us, be pronounced satisfactory.

Repeated analyses of the same sample and on two different samples of each fodder made by the reporter on foods low in fiber gave practically the same percentages as put down in the table, and the low result can not therefore be attributed to faulty determination or to imperfect sampling of the feeds sent out. The apparatus used in the Wisconsin work is a small, rectangular, double-walled, copper oven, in which the fodder is placed in small glass disks; dry hydrogen gas is conducted into the oven and passes out at the opposite side along with the moisture and the volatile matter of the samples. Cutter's results (Bull. 6, Cornell Univ. Exp. Station, June, 1889) showed that a greater loss takes place in heating the sample in a tube in a current of air than in open watch glasses, the temperature being the same in both cases. The reason for this may be that the hydrogen can not perfectly replace the air in the latter case, and that we therefore have practically the effect of heating in air. From the data given in the table, as well as from the results of many earlier investigations (Jenkins, Atwater, Baessler, Anderson, Cutter, Van Slyke, Woll, and others), we know that the loss occurring in heating a fodder is greater in a medium of hydrogen than in the air. None of the analysts reporting stated their method of drying but Mr. Huston, beyond saying that the official method was followed. Mr. Huston's results were obtained by drying in hydrogen for four hours at 100° C. in an apparatus similar to Fig. 11, p. 100, Bull. No 28, U. S. Department of Agriculture, division of chemistry. The late date at which most of the reports were received made it impossible to ascertain the kind of apparatus used by each chemist, but it seems very probable that differences in these is the main cause of the variations in results obtained.[1]

Another cause, doubtless, is the time of drying; the official method says five hours. It will depend on the method of drying, however, whether or not complete desiccation will take place within this time. Where hydrogen is conducted over and not through the samples the five-hour limit will as a rule be found too short; where hydrogen is conducted through the samples the velocity of the current will determine the time of drying. (Anderson Bull. 35, Chem. Div., 132.) In the drying oven used by the reporter on foods low in fiber constant weight can not be obtained short of seven to ten hours, according to the number of samples placed in the oven at a time. In drying the samples of corn meal and oil meal the following data were obtained, each figure given being the average of four determinations:

Percentage loss in drying samples of corn meal and oil meal.

Article.	5 hours.	7 hours.	8 hours.	9 hours.	10 hours.	11 hours.
Corn meal	8.39	8.96	9.35	9.53	9.55	9.44
Oil meal	6.42	6.86	7.40	7.49	7.58	7.45

[1] Since the above was written, the reporter has learned that in the determinations by Messrs. Kilgore and Voorhees hydrogen was conducted over the samples in drying, which hydrogen was passed through the samples in the determinations made by Messrs. Rossman, Runyan, Teller, Huston, Shiver, and Patterson.

ETHER EXTRACT.

The investigations of methods of ether extraction made by the various chemists are fairly complete, as will be seen from the following table:

Methods of ether extraction.

CORN MEAL.

Analysts.	Samples dried in—		Sample air-dry.	Charcoal method.
	Hydrogen.	Air.		
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1. Patterson, Mississippi	4.53	4.41	4.47	3.47
2. Shiver, South Carolina	4.37	4.18	....	....
3. Kilgore, North Carolina	4.06	4.20	4.34	3.80
4. Rossman & Harrison, Michigan	4.24	....	....	....
5. Voorhees, New Jersey	4.30	....	4.28	....
6. Bartlett, Maine	4.19	4.18	4.34	4.03
7. Runyan, U. S. Department of Agriculture	4.45	....	4.51	3.99
8. Teller, Arkansas	4.55	....	4.60	4.35
9. Woll, Wisconsin	4.29	....	4.68	3.87
Average	4.33	....	....	....
Greatest variation from average	.27	....	....	....
Greatest variation from average in per cent	6.2	....	....	....
Difference between highest and lowest	.49	....	....	....
Difference between highest and lowest in per cent	11.30	....	....	....
Average of 1, 2, 3, and 6	4.29	4.24	....	....
Average of 1, 3, 5, 6, 7, 8, and 9	4.34	....	4.46	....
Average of 1, 3, 6, 7, 8, and 9	4.35	....	....	3.92

OIL MEAL.

1. Patterson, Mississippi	6.87	6.65	6.74	4.92
2. Shiver, South Carolina	6.77	6.83	....	....
3. Kilgore, North Carolina	6.83	6.73	7.10	6.18
4. Rossman & Harrison, Michigan	6.69	....	....	....
5. Voorhees, New Jersey	6.70	....	6.77	....
6. Bartlett, Maine	6.65	6.58	6.70	6.11
7. Runyan, U. S. Department of Agriculture	6.71	....	6.79	6.22
8. Teller, Arkansas	6.90	....	7.05	6.76
9. Woll, Wisconsin	6.86	....	6.88	5.69
Average	6.78			
Greatest variation from average	.13	....	....	....
Greatest variation in per cent	1.90	....	....	....
Difference between highest and lowest	.25	....	....	....
Difference between highest and lowest in per cent	3.70	....	....	....
Average of 1, 2, 3, and 6	6.78	6.70	....	....
Average of 1, 3, 5, 6, 7, 8, and 9	6.79	....	6.86	....
Average of 1, 3, 6, 7, 8, and 9	6.80	....	....	5.98

REMARKS ON ETHER EXTRACT AND SUPPLEMENTARY INVESTIGATIONS BY ANALYSTS.

Mississippi.—Mr. Patterson writes: "The fat in the animal-charcoal method falls so far below that obtained by the official method that I suspect that the charcoal not only retains the coloring matter, etc., but some of the fat, also."

North Carolina.—"The time of extraction was sixteen hours in all cases, though quite a number were reëxtracted for twenty-four hours more after the first weighing, with little or no change in the amount of extract. The results by the charcoal method are not altogether satisfactory to myself. I prefer the two higher results as representing more nearly the content of oil in corn meal (single determinations, for corn meal, 3.64, 3.61, 3.98, 3.98; for oil meal, 6.23, 6.13); but why the others should have fallen so low I can not say, as the manipulation was practically the same in all cases. The 6.23 result on the oil meal was obtained with the meal in contact with the charcoal, but in all other cases the meals were separated from the charcoal by a thin layer of extracted cotton, 0.9 to 1.15 grams charcoal was used."

The extracts from each of the meals by each of the four methods were combined, treated with water and a few drops of HCl, heated on water bath, and tested with Fehling solution for reducing sugars, with the following results:

(1) After drying and with charcoal, a very slight reduction.
(2) After drying and without charcoal, a slight reduction.
(3) Without drying and without charcoal, a considerable reduction.
(4) Without drying and with charcoal a slight reduction.

The reduction was more decided with the oil meal extract than with the corn meal, and was decidedly heaviest in "Method No. 3."

New Jersey.—Mr. Voorhees reports the following results in addition to those given in the table:

	Corn meal.			Linseed meal.		
	Ether dried by sodium.	Ether dried by CaCl$_2$.	Ether saturated with water.	Ether dried by sodium.	Ether dried by CaCl$_2$.	Ether saturated with water.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Dried in hydrogen five hours	[1]4.30	4.28	....	[1]6.70	6.73	....
Undried	4.28	4.28	4.09	6.77	6.75	6.74

[1] The results were secured with the top of the condensing apparatus protected by a CaCl$_2$ tube to exclude moisture.

Maine.—Animal-charcoal method.

Samples not dried.

	1 gram charcoal.	2 grams charcoal.	No charcoal.
	Per cent.	*Per cent.*	*Per cent.*
Extracted sixteen hours:			
Sample 1	....	4.03	4.34
Sample 2	6.35	6.11	6.70
Extracted six hours longer:			
Sample 1	....	4.16	....
Sample 2	6.59	6.36	....

A few tests were made to see how readily pure oils could be extracted through charcoal. Extracted for sixteen hours:

	1 gram charcoal.		2 grams charcoal.	
	Weighed out.	Extracted.	Weighed out.	Extracted.
	Gram.	*Gram.*	*Gram.*	*Gram.*
Cotton-seed oil	.9427	.9117	.8995	.8602
Linseed oil	.9933	.9042	1.0536	.8162
Average loss	....	.0601	....	.1414

Since doing the above work, it has occurred to me that there may be a difference in charcoals; some may retain fats and others may not. My experience with the charcoal method for purifying fats is similar to yours of last year; I could not recover all the oils in a reasonable time. I have become quite well convinced that drying in hydrogen has no advantages over drying in air.

Arkansas.—I tried ether extractions on samples of corn meal, wheat bran, and oil meal, dried in hydrogen, and afterwards remoistened by pouring through them a stream of moist hydrogen, with results as follows:

	Not dried.	Dry.	Remoistened.
	Per cent.	*Per cent.*	*Per cent.*
Corn..............................	4.50	4.39	4.30
Wheat bran (shorts)..................	4.45	3.95	4.75
Linseed oil meal.....................	7.05	6.90	6.92

"The figures given are the average of duplicates. On the bran the results agree closely with those you obtained on samples dried and not dried, as reported in the association report of last year. I think the results on this point show that the effect of previous drying depends on the nature of the materials under analysis. Both the remoistened bran and linseed contain a little more moisture than before treatment."

New York (Geneva).—Mr. C. G. Jenter has made a large number of extractions to determine whether or not charcoal retains fat from ethereal solutions. The method of procedure is described by Mr. Jenter as follows:

"In both methods the fat was absorbed by a small piece of cotton and extracted with anhydrous, alcohol-free ether. Two grams of charcoal were used in all cases in the charcoal methods. The charcoal and cotton were previously extracted for about thirty hours, yet it was found that they still gave up some extract, with ether, even after being extracted for fifty hours or more, and thus several blanks were carried through with each set and the amount deducted from the total extract.

"The charcoals used in 1, 2, and 4 were coarse animal charcoal which would pass through a No. 20 sieve, while that used in Table No. 3 was powdered and passed through a No. 50 sieve in order to see if any of the fat was retained in the pores of the coarser charcoal. The fat and the tubes were dried in a water jacketed air oven, temperature 98°."

The following tables give the results of the work done.

Butter fat (not butter).

	Weight taken.	Official.		Weight taken.	Charcoal.	
		Direct.	Indirect.		Direct.	Indirect.
Extracted six hours........	.9178	—.0038	—.0013	.8419	—.0156	—.0027
	1.1428	+.0012	—.0007	.9174	—.0119	—.0005
	.8450	—.0015	—.0007	.7971	—.0123	—.0040
	.6837	—.0037	—.0001	.6279	—.0081	+.0013
	.3272	—.0042	+.0003	.5010	—.0076	—.0023
	.3968	—.0028	+.0016	.7034	—.0200	—.0119
	.4020	—.0007	+.0003	.3843	—.0112	—.0051
	.3062	—.0011	+.0005	.3334	—.0063	—.0033
	.4716	+.0004	—.0007	.4510	—.0127	+.0025
	.4309	—.0018	—.0001	.3913	—.0009	+.0005
	.3390	+.0009	—.0005	.3780	—.0056	—.0060
	.3253	—.0005	—.0001	.3176	—.0059	—.0016
	....	....	....	.2925	—.0003	—.0059
	....	....	....	.5136	—.0318	—.0036
Average................	....	—.00146	—.00012	....	—.01073	—.00304

Butter.

	Amount taken.	Official.		Amount taken.	Charcoal.	
		Direct.	Indirect.		Direct.	Indirect.
Extracted fourteen hours ..	.4998	+.0021	—.0003	.5655	—.0087	—.0073
	.3902	+.0018	—.0002	.3214	—.0051	—.0093
	.4689	—.0012	±.0000	.3140	—.0036	—.0067
	.4220	—.0055	+.0002	.5757	—.0057	—.0001
	.4000	—.0014	+.0006	.5264	—.0067	—.0100
	.3826	—.0055	+.0003	.5533	—.0062	—.0707
	.5142	+.0009	+.0005	.6512	—.0030	—.0090
	....	....	....	.4822	—.0036	—.0068
	....	....	....	.4785	—.0121	—.0090
	....	....	....	.6199	—.0156	—.0048
Average................	....	—.00125	+.00016	....	—.00703	—.00731

Butter.

	Amount taken.	Charcoal.			Amount taken.	Charcoal.	
		Direct.	Indirect.			Direct.	Indirect.
Ext. 14 hours	.3366	—.0179	—.0120	Ext. 14 hours	.5623	—.0198	—.0122
	.4995	—.0289	—.0130		.6659	—.0188	—.0066
	.6822	—.0194	—.0095		.6650	—.0192	—.0066
	.6009	—.0173	—.0103		.6690	—.0185	—.0073
	.5389	—.0083	—.0038		.6637	—.0233	—.0109
	.6718	—.0201	—.0102		.5337	—.0163	—.0056
	.5496	—.0213	—.0115		.4860	—.0160	—.0073
	.6074	—.0244	—.0111		.6180	—.0244	—.0070
	.4983	—.0208	—.0106		.5838	—.0020	—.0020
	.4298	—.0164	—.0095				
	.5204	—.0138	—.0065	Average	....	—.01746	—.00869
	.5929	—.0198	—.0070				

Lard.

	Weight Taken.	Official.		Weight taken.	Charcoal.	
		Direct.	Indirect.		Direct.	Indirect.
Extracted twelve hours.....	.3427	+.0030	—.0004	.4616	—.0034	—.0016
	.3166	+.0027	—.0004	.5683	—.0041	—.0023
	.4307	—.0002	+.0003	.5767	—.0022	—.0001
	.5628	+.0014	+.0011	.4221	+.0047	—.0005
	.6000	—.0011	+.0003	.3124	—.0029	—.0004
	.5408	—.0001	+.0002	.6651	—.0021	+.0028
	....	....	....	.3588	±.0017	—.0010
	....	....	....	.3973	±.0000	—.0002
	....	....	....	.5208	—.0027	+.0010
	....	....	....	.4014	—.0026	+.0004
Average	....	+.00095	+.00018	....	—.00136	.00019
Extracted fourteen hours...	.5945	—.0012	—.0002	.4024	—.0036	—.0044
	.4465	—.0021	—.0006	.4689	—.0018	—.0026
	.3659	—.0005	+.0004	.3995	—.0058	—.0034
	.5141	+.0012	—.0003	.5955	—.0025	—.0011
	.3839	—.0022	—.0001	.5118	—.0015	—.0024
	.3546	—.0015	—.0001	.2443	+.0003	—.0002
	.5124	—.0031	+.0023	.4554	—.0008	—.0016
	....	....	....	.5172	—.0041	—.0006
	....	....	....	.5849	—.0056	—.0013
Average.................	....	—.00134	+.0002	....	—.00282	—.00195

COMMENTS ON ETHER EXTRACT.

Official method.—The results obtained by nine different analysts are very concord-ant, the widest difference being 0.49 per cent for the corn meal and 0.25 per cent for the oil meal; calculated on the average per cent of ether extract the greatest varia-tions from average amounts to 6.2 and 1.9 per cent for the corn meal and oil meal, respectively, which is greatly below the 10 per cent limit adopted by Prof. Caldwell.

The effect of drying samples in the air previous to extraction.—Four analysts give data for extraction of samples dried in hydrogen and in the air; the average results of the air-dried samples are lower by 0.05 and 0.08 per cent for corn meal and oil meal, respec-tively. This is in accord with the results obtained in most earlier investigations of the subject (Maercker, Wilm, Buhring, Baessler, Anderson, Farrington, Woll, and others). While the differences found in this case are not large, and sometimes go in the opposite direction, they would be greatly increased where the feeds are heated above 100° C., or for a longer time than five hours. The food stuffs containing a large amount of oil will give the lowest results on drying in air previous to extrac-tion, the decrease being due to oxidation and partial change of the fat into ether-insoluble resinous compounds. The results in case of samples dried in the air pre-vious to extraction may be correct in many cases, but the method can not be used universally, and can not, therefore, be recommended in preference to drying in hydro-gen previous to extraction.

The effect of extraction without drying.—The results obtained by seven chemists in comparative determinations are higher on an average for samples not dried than for those dried in hydrogen. This is as we should expect, and is in accordance with pre-vious work in this line. The water in the sample will in case of certain feed stuffs dis-solve out part of the water-soluble constituents of the sample and thus increase the

ether extract (see the remarks of Messrs. Kilgore and Teller in the above). The excess dissolved out is of course dependent on the amounts of water and of water-soluble constituents of the sample.

There are several reasons for believing that the process of drying, be it in hydrogen or in air, even at the temperature of 100° C., modifies the results of ether extraction to some extent, and it has therefore repeatedly been suggested that moisture and ether extract ought to be determined in different portions of the fodders. As extraction without previous drying gives too high results, it has occurred to the reporter on foods low in fiber that the copper-sulphate method, as originally devised by Morse and Piggot for the estimation of fat in milk (Am. Chem. Jour. 9, 108), might be used with advantage for this purpose. Although only very little analytical work has been done so far in ascertaining the correctness and adaptability of the method for fodders, we give it here, as some chemists may be willing to try it during the coming year.

From 2 to 3 grams of the fodder are weighed out on a watch glass; about 5 grams of anhydrous copper sulphate are added and mixed with the sample; the whole is then transferred to an extract iron tube and the extraction proceeded with as usual.

The following results were obtained with the samples sent out by the reporter:

	Corn meal.	Oil meal.
	Per cent.	*Per cent.*
Ether extract by official method (anhydrous ether)	4. 29	6. 88
Ether extract by new method (washed ether)	4. 34	6. 88

The duplicates by the copper-sulphate method agreed within a tenth. The advantages of the new method are, first, there is no drying to influence the solubility of the fat, and, second, any ether may be used, washed, concentrated, or absolute, as the sulphate at once takes up any water that may be in the ether. The possibility of a doing away with the very particular process of dehydration of the ether by sodium gives, I am free to say, a good deal of charm to the method for me. Another advantage may be that extraction in this case possibly can be completed in less time than the official sixteen hours; but that is theory.

The animal charcoal method.—This was investigated by five chemists. The results in all cases came lower, as we should expect and as others have found. The method stands or falls with the question, whether or not animal charcoal retains pure fat from ethereal solutions. Mr. Jenter's extensive work in this line forms a most important contribution. The results are in accord with those of the reporter on foods low in fiber, published in Bulletin 35 of the Chemical Division. The losses of fat in the indirect determinations would seem to show that the charcoal used was not perfectly dry; but the minimum effect of the charcoal may be obtained by subtracting the losses found in the official method from those found in the charcoal method. The following table gives the losses and differences:

	Official method.	Charcoal method.	Differences.
Average losses in extraction:	*Grams.*	*Grams.*	*Grams.*
Butter fat (14 determinations)	. 00146	. 01073	. 00927
Butter fat (10 determinations)	. 00125	. 00703	. 00570
Butter fat (21 determinations)	....	. 01746	....
Lard (10 determinations)	. 00095	. 00136	. 00041
Lard (9 determinations)	. 00134	. 00282	. 00148
Total and averages (64 determinations)	. 00125	. 00588	. 00463

The average figures obtained by one of us (Woll), as given in Bulletin 35, p. 126, of the U. S. Department of Agriculture, Division of Chemistry, were: Average for 15 determinations, .00528.

The close agreement between these average losses in the charcoal method may show that the amount of fat taken up by a certain quantity of a sample of charcoal is fairly constant, but the results of Mr. Barlett, given above, would tend to disprove any such proposition. The quantity of charcoal is of importance, and also its quality. Mr. Bartlett's figures illustrate the former point, and also those given in the following table (see, also, Remarks on Ether Extract, under *Maine*, p. 125):

Analyst.		Corn meal.	Oil meal.
		Per cent.	*Per cent.*
Runyan............	1 gram animal charcoal....................	3.99	6.22
	2 grams animal charcoal	3.93	6.08
Woll..............	1 gram animal charcoal	3.87	5.69
	2 grams animal charcoal	3.42	5.14

The kind of charcoal is also important, as some kinds will take up more fat than others. The chemists investigating the method all received their charcoal from the reporter on foods low in fiber, so that the variations in results can not be ascribed to a difference in charcoal; but the following results obtained on two samples of charcoal, both C. P., but treated alike, will illustrate this. The figures can only be compared with one another vertically:

	Corn meal.			Oil meal.		
	I.	II.	III.	I.	II.	III.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Charcoal A	3.27	3.19	4.00	5.76	4.62	5.71
Charcoal B.................	3.87	3.65	4.27	5.69	5.66	6.26

The animal-charcoal method for purification of ether extract was first proposed by G. Kuehn about twenty-five years ago (Wolff, Unters. landw. wicht. Stoffe, 2nd ed., 142), and König, in 1871, showed that it was not reliable, having obtained the following results (L. Vers. Stat. **13**, 242):

	Weighed out.	Regained.	Loss.
Fat from—			
White sesame....................	2.0893	2.0027	.0866
	1.8854	1.8036	.0818
Beechnuts........................	1.6120	1.4483	.1637
Oat straw........................	.4145	.3861	.0284

König's results were confirmed by Schulze in 1872 (L. Vers. **5**, 81). The method was brought forward again a few years ago and has been discussed in several meetings of our association. In view of the data given in the above, your reporter can not recommend it as a quantitative method unless each sample of charcoal used be tested by careful experiments previous to its use, and a correction made; judging from work done by Jenter and Woll, the minimum error introduced by the charcoal would amount to a quarter of one per cent for 2 grams of substance.

Crude fiber.—Seven reports were received on the determination of crude fiber by the official method and four by Hönig's method; the results are given in the following table:

Determination of crude fiber.

Analyst.	Hönig's method.		Official method.	
	Corn meal.	Oil meal.	Corn meal.	Oil meal.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1. Kilgore, North Carolina.............	3.17	17.90	1.43	7.35
2. Huston, Indiana....................	7.84	18.56	1.73	7.85
3. Shiver, South Carolina.............	....	....	1.48	6.27
4. Nossman and Harrison, Michigan......	....	....	1.59	8.03
5. Voorhees and Street, New Jersey.....	1.70	11.59	1.37	6.71
6. Teller, Arkansas....................	....	....	1.69	7.50
7. Runyan, U. S. Department of Agriculture......	4.50	19.09	1.57	7.09
Averages............................	....	....	1.55	7.26
Greatest variation from average........	....	....	.18	.99
Greatest variation in per cent of average..........	....	....	11.6	13.6
Difference between highest and lowest result......	....	....	.36	1.76
Difference between highest and lowest in per cent of average........	....	....	23.2	24.2

Three chemists reported determinations of crude fiber by 1.25 and 2.50 per cent solutions, with the following results:

Official method of crude fiber determination.

Analyst.	Corn meal.		Oil meal.	
	1¼ per cent solution.	2½ per cent solution.	1¼ per cent solution.	2½ per cent solution.
Huston, Indiana....................	1.89	1.73	8.21	7.85
Teller, Arkansas....................	1.77	1.69	8.57	7.50
Voorhees and Street, New Jersey......	1.46	1.37	7.60	6.71
Averages.........................	1.71	1.60	8.13	7.35

REMARKS ON CRUDE FIBER AND ADDITIONAL INVESTIGATION.

North Carolina.—The fiber residues of both the meals by both the Hönig and the official methods were tested in duplicate (quantitatively) for pentosans, which were present in all cases, though the amount was very small in case of the corn meal by the official method, but more decided by the Hönig method. The fiber from the oil meal evidently contained a considerable amount of pentosans, especially that obtained by the Hönig method. The anilin-acetate test for pentosans was employed, boiling the fiber residue in HCl (sp. gr. 1.06). The nitrogen and ash found in the residue were as follows:

	Corn meal.		Oil meal.	
	Hönig method.	Official method.	Hönig method.	Official method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Ash........................	.50	.24	2.87	.25
Nitrogen.....................	.04	.03	.34	.10

Indiana.—Only slightest trace of pentosans found in the original material and none in the alleged fiber. The following percentage of ash and albuminoids were found in the residues:

	Corn meal.			Oil meal.		
	Hönig method.	Official method.		Hönig method.	Official method.	
		1¼ per cent solution.	2½ per cent solution.		1¼ per cent solution.	2½ per cent solution.
Ash	.63	.05	.03	3.28	.28	.08
Albuminoid solution......	1.27	.05	.04	3.52	.05	.05

Prof. W. E. Stone has kindly furnished me the following directions for a qualitative examination of vegetable fibers for pentosans:

Test for pentosans.—Apparatus and chemicals needed: a narrow deep beaker or large test tube; HCl of 1.06 sp. gr. or about 12 per cent strength; strong acetic acid, and commercial anilin.

The fiber is boiled in the beaker or test tube with a small quantity of HCl; the exact proportion is not important. Equal portions of the acetic acid and anilin are mixed, and warmed if the mixture is not complete (4 or 5 cc. of the reagent will be sufficient for many tests). A slip of filter paper is moistened with anilin acetate and held in contact with the vapor coming off from the boiling fiber and HCl. A small amount of furforol in this vapor (which can only come from pentosans) will impart a bright crimson color to the paper. If such a reaction is not obtained within four or five minutes, no pentosans are present; it usually appears within a minute. The test is as reliable as it is simple.

New Jersey.—The determinations by the association method were made with 1.25 per cent acid and alkali, and 2.50 per cent also, as a matter of experiment.

The ash and nitrogen in the fiber by Hönig's method were determined in one instance each, amounting to as follows:

	Ash.	Nitrogen.
	Per cent.	*Per cent.*
Corn meal	1.25	.05
Oil meal	1.89	.36

In the results above reported the ash has been deducted, but not the nitrogen.

U. S. Department of Agriculture.—The results obtained by Hönig's method are not considered reliable. They were obtained on a preliminary trial of the method and apparatus. The residues gave reactions for pentosans by Stone's test (anilin acetate).

Correction was made for ash in the fiber residues, but not for nitrogen. In one case the residue from corn meal gave .55 per cent ash, calculated on weight of sample, and one residue from the linseed meal gave 2.94 per cent ash. The amount of nitrogen was not determined.

COMMENTS ON RESULTS OF CRUDE FIBER DETERMINATION.

Official method.—In the official method the highest and lowest per cent found came .36 per cent and 1.76 per cent for corn meal and oil meal, respectively, or 23.2 and 24.2 per cent of the average crude-fiber content in the two cases. While the result is as good as could be expected for the corn meal, this is not the case with the oil meal.

The table giving the results of the treatment with solutions of different strength shows that the stronger solutions have a more powerful action on the crude fiber than the weaker ones. We shall come back to this point farther on.

Hönig's method.—Your reporters have not had the presumptuousness to calculate the averages of the figures given in the table for Hönig's method. The results with the method as found by four different analysts are certainly a surprise, and, as it would seem, strongly condemn the method. When experienced chemists, all of whom are more or less specialists in fodder analysis, obtain percentages varying from 1.70 per cent to 7.84 per cent in the case of corn meal, and from 11.59 to 19.09 per cent in the case of oil meal, then the method must be at fault. It is true that it is comparatively new, and, as far as we know, was never worked before by any of the analysts, but they all had the benefit of the experience of Hönig, Gabriel, and Jordan with the method, and were doubtless able to conquer the main difficulties of manipulation. It will be noticed that the nitrogen and ash left in the fiber is very considerable and must be corrected for. Corrections were not made for nitrogen in case of Mr. Runyon's determinations, which at least partly explains the high per cent found for the oil meal.

Feed stuffs high in fiber.—The reporter on foods and feeding stuffs high in fiber in the beginning of April last sent out six samples of feeds to chemists, wanting to undertake a study of crude fiber determinations, the investigations being intended to search for the proper line, if any, for the division made by the association, in foods rich and foods poor in fiber. Only one chemist besides the reporter himself found it possible to do the work outlined, and the report therefore covers only two sets of determinations. The samples sent out are given in the table with the results obtained:

Summary of crude fiber determinations by Weende method.

	Indiana Station.			Arkansas Station.		
	2½ per cent solutions.	1¼ per cent solutions.	Difference.	2¼ per cent solutions.	1¼ per cent solutions.	Difference.
Millet seed	7.21	8.87	1.66	7.17	8.76	1.59
Pea vine..................	21.26	24.08	2.82	21.32	24.30	2.98
Clover seed..............	10.00	11.35	1.35	8.87	9.81	.94
Timothy hay	28.32	29.90	1.58	29.56	32.14	2.58
Corn bean	12.60	13.20	.60	12.96	13.73	.77
Corn husks...............	27.50	29.70	2.20	27.25	30.70	3.45
Averages............	17.82	19.52	1.70	17.86	19.91	2.05

An inspection of the above results will show that in every case a lower result was obtained by the treatment of the stronger solutions. In view of this, and further on account of the lack of other analytical work in this line, your reporters consider it of importance that the question of the strength of the solutions be taken up and studied during the coming year. It seems probable, judging from all determinations made by the Schulze and the Hönig methods, that an appreciable quantity of fiber is dissolved by the action of the solutions in the Weende (*i. e.*, the official) method; to increase this quantity further by the application of stronger solutions does not seem good policy to your reporters. As far as ease of manipulation[4] is concerned, Mr. Teller reports that he could find no great difference with the two strengths of solutions in none of the samples worked, including those of corn meal and linseed oil. A sample of patent wheat flour would not give a clear solution, however, even with 2½ per cent acid and alkali. The milkiness of the filtrates in this case was probably due to undissolved starch. The nitrogen and starch determinations did not show any marked improvement of the one method over the other, in so far as they are concerned.

The following complete analyses of the feed stuffs sent out by the reporter on foods low in fiber were received:

Analyses of corn meal and oil meal.

	Moisture.	Ash.	Crude protein.	Crude fiber.	N–free extract.	Ether extract.
Corn meal:						
Shiver, South Carolina......	10.20	1.65	9.84	1.48	72.46	4.37
Rossman & Harrison, Michigan....................	10.52	1.69	9.41	1.59	72.55	4.24
Teller, Arkansas...........	10.30	1.55	9.81	1.69	72.10	4.55
Averages.................	10.34	1.63	9.69	1.59	72.36	4.39
Linseed meal:						
Shiver, South Carolina.....	8.42	5.57	37.03	6.27	35.94	6.77
Rossman & Harrison, Michigan.......................	8.47	5.92	36.69	8.03	34.20	6.69
Teller, Arkansas...........	8.85	5.47	37.12	7.50	34.16	6.90
Averages.................	8.58	5.65	36.95	7.27	24.76	6.79

	Corn meal.			Oil meal.		
	Total N.	Alb. N.	Amide N.	Total N.	Alb. N.	Amide N.
Rossman & Harrison, Michigan	1.51	1.50	.01	5.87	5.37	.50
Teller, Arkansas	1.57	1.53	.04	5.94	5.21	.73

The results for moisture, crude fiber, and ether extract have already been discussed in the preceding; the data on hand for the other components are too few to warrant any conclusions to be drawn from the results.

RECOMMENDATIONS.

Your reporters on cattle foods are of the opinion that the division of the methods of fodder analysis between two reporters is unfortunate and against the best interests of the association. The divisional line is arbitrary and indefinite and has been a constant source of complications and misunderstandings during the years in which it has been in force; one chemist will take a sample of fodder for high in carbohydrates, which another will class under those low in carbohydrates. The reporter on foods low in fiber considered linseed meal as belonging under his domain, while the other reporter placed it under his class. Difficulty arises in planning the work and a still greater difficulty comes in the possibility of multiplication of the work undertaken. It is therefore hard to see the justification for a division. One man can better plan the work to be done than two, especially since there is never time for the two reporters to confer with one another previous to sending out samples; further, the station chemist ought not to be asked to do more work in investigating methods than is strictly necessary in order to fulfill the ends of the association.

Nitrogen determinations are made by a different method in samples containing nitrates than in those containing organic or ammoniacal nitrogen, but one reporter is considered able to handle the whole subject. The same holds good in potash analysis, ash analysis, sugar analysis, etc. In the analysis of cattle foods a division is made in the crude fiber determinations, with all the other determinations common for both reporters. While we are not yet ready to say that there is no ground for a division of methods in crude fiber determinations, we are of the opinion that the whole subject of fodder analysis ought to be left with one reporter, who can

then plan the investigational work along the exact lines where it is most needed, and with such representative feeds as may best serve to illustrate the objects sought.

Your reporters may be pardoned for stating that in several respects the present scheme of fodder analysis is in their opinion highly unsatisfactory, especially as regards the determinations of crude fiber and nitrogen-free extract; the latter heterogeneous class of bodies often make up more than one-half of the fodders, and contains within it ingredients of the highest value in nutrition as well as such as can have hardly any value at all. We are aware that comparable results for both groups are obtained by a strict observance of the directions laid down, but this does not prove their correctness. While your reporters do not deem it advisable to propose a new scheme of analysis in the place of the present methods, we would suggest that this subject be discussed by the association, and that methods of determination of sugar and starch be studied with a view of including these components in the analyses. With sugar, starch, crude fiber, and possibly pentosans determined in a fodder, we could afford to place the organic nonnitrogenous and nonfatty rest as *undetermined*, and in our opinion we would be then in far better condition to judge about the intrinsic value of the various feeding stuffs than we are at the present. While similar objections may be justly made to other groups of components determined in our present methods, we believe that none need more careful and immediate study than the so-called carbohydrates.

Your reporters would offer the following specific recommendations:

(1) The subject of cattle foods is to be assigned to one reporter.

(2) The official method for moisture is to be changed to read: Dry 2–3 g. of the substance at the temperature of boiling water for five hours by passing a current of dry hydrogen through the sample.

Mr. DE ROODE moved the adoption of the recommendations made by the reporter.

The PRESIDENT. Before putting that motion to a vote I think it would be well to present any other papers (I believe there are one or more) bearing on the subject of fodder analysis.

Mr. HUSTON read the following paper:

PRELIMINARY INVESTIGATIONS RELATING TO THE DETERMINATION OF CRUDE FIBER.

By H. A. HUSTON and F. W. MCBRIDE.

The work reported below contains the results so far obtained in investigating the processes and products connected with the determination of "crude fiber" in feeding stuffs. The work is still in progress, and while much of the work included in the original plan is unfinished, it was thought best to report the results so far obtained as they have a direct bearing on the present and proposed methods of the association.

The results obtained are divided into three sections:

(1) Action of various solvents on the samples sent out by the association reporters on feeding stuffs. A sample of clover hay used for trial work is also included.

(2) Action of these solvents on Schleicher & Schüll filter paper No. 589.

(3) Action of solvents for cellulose, vasculose, and lignose on the fiber resulting from the official method and from treatment with dilute sulphuric acid and glycerol.

The solvents used were the 2¼ and 1¼ per cent sulphuric acid and caustic soda of the official methods, glycerol, a 4 per cent solution of borax, a solution of silicate of soda sp. gr. 1.0425, 25 cc. of which neutralized 19.75 cc. one-half normal hydrochloric acid, made by diluting 225 cc. commercial silicate of soda to 2000 cc. Carbonate of soda, solution neutralized same amount of one-half normal HCl as 2¼

per cent NaOH, bicarbonate of soda, solution neutralized same amount of one-half normal HCl as 2½ per cent NaOH, hydrochloric acid 2½ per cent solution and distilled water.

RESULTS ON FEEDING STUFFS SENT BY REPORTERS.

The feeding stuffs were extracted with ether before treatment with the solvents with the exception of the material used in Hönig's method and in No. 3, where a set of special determinations were made without extraction with ether. All digestions were continued for thirty minutes at the boiling point unless otherwise stated in the table. Two hundred cc. of fluid were used with the exception of glycerol, of which 60 cc. were used. Two grams of original material were used throughout.

TABLE I.—*Results of various solvents on feeding stuffs sent out by association reporters.*

Method used.	Unhulled millet seed.				Whippoorwill pea vine.				Red clover seed.			
	Total residue.	Ash.	Albuminoids.	Fiber.	Total residue.	Ash.	Albuminoids.	Fiber.	Total residue.	Ash.	Albuminoids.	Fiber.
	Grs.	Grs.	Grs.	Per cent.	Grs.	Grs.	Grs.	Per cent.	Grs.	Grs.	Grs.	Per cent.
1. Official 1¼ per cent H_2SO_4 and 1¼ per cent NaOH.	.1900	......	.0103	8.98	.5750	......	.0000	24.10	.2340	.0052	......	11.02
	.1855	.0055	......	8.76	.5745	.0930	......	24.07	.2475	......	.0073	11.70
2. Official 2½ per cent H_2SO_4 and 2½ per cent NaOH.	.1445	......	.0009	7.18	.5365	......	.0003	21.32	.2030	.0015	......	9.92
	.1460	.0000	......	7.25	.5340	.1070	......	21.20	.2055	......	.0030	10.05
3. 2½ per cent H_2SO_4 and 2½ per cent NaOH, but material not extracted with ether before treatment.	.1780	.0040	......	8.70	.5280	.0920	......	21.80	......	......	......	......
	.1330	.0065	......	6.32	.5260	.0950	......	21.55	......	......	......	......
	.1660	.0050	......	8.05	......	......	......	......	......	......	......	......
	.1595	.0020	......	7.87	......	......	......	......	......	......	......	......
	.1580	.0020	......	7.80	......	......	......	......	......	......	......	......
4. Hönig's method.	.5495	.0460	......	25.17	.9530	.1815	......	35.40	.4755	.0380	......	20.83
	.5645	......	.0000	25.92	.8400	......	.0837	29.75	.4475	.0355	......	19.43
	......	......	......	......	.8470	......	......	30.10	.4820	......	.0222	21.15
	......	......	......	......	.8800	.1410	......	31.75	......	......	......	......
5. 1¼ per cent H_2SO_4.	.5515	......	......	......	1.0030	......	......	......	.5840	......	......	......
	.5680	......	.1359	......	1.0130	......	.1442	......	.5540	......	......	......
	.4625	......	......	......	.9930	......	......	......	.5980	......	.0966	......
6. 1¼ per cent H_2SO_4 followed by glycerol, etc., as in Hönig's method.	.2760	......	.0046	12.47	.6520	.0980	......	27.03	.3685	.0070	......	16.50
	.2520	.0220	......	11.27	.6650	......	.0133	27.68	.3760	......	.0314	16.88
7. 1¼ per cent of H_2SO_4 and 4 per cent borax.	.3390	.0055	......	12.35	.7310	.0635	......	29.67	.3265	......	.0315	14.42
	.3440	......	.0875	13.10	.7690	......	.0748	31.53	.3031	.0065	......	13.25
8. 1¼ per cent H_2SO_4 and silicate of soda.	.3150	.0190	......	11.33	.7390	......	.0490	28.32	.2800	.0140	......	12.38
	.3260	......	.0694	11.88	.7170	.1235	......	26.72	.3040	......	.0184	13.58
9. 1¼ per cent H_2SO_4 and carbonate of soda.	......	......	......	......	......	......	......	......	......	......	......	......
10. 1¼ per cent H_2SO_4 and bicarbonate of soda.	......	......	......	......	......	......	......	......	......	......	......	......
11. Borax 4 per cent, 1 hour.	.8510	.1175	......	30.14	1.0256	.1565	......	35.60	.9410	......	.1676	35.87
	.6980	......	.1306	22.49	1.0520	......	.1570	36.92	.9250	.0560	......	35.07

TABLE I.—*Results of various solvents on feeding stuffs sent out by association reporters—* Continued.

Method used.	Timothy hay.				Corn bran.				Corn husks.			
	Total residue.	Ash.	Albuminoids.	Fiber.	Total residue.	Ash.	Albuminoids.	Fiber.	Total residue.	Ash.	Albuminoids.	Fiber.
	Grs.	Grs.	Grs.	P. ct.	Grs.	Grs.	Grs.	P. ct.	Grs.	Grs.	Grs.	P. ct.
1. Official 1¼ per cent H_2SO_4 and 1¼ per cent NaOH.	.6120	......	.0030	30.10	.2675	......	.0012	13.29	.6000	.0055	......	29.68
	.6080	.0090	......	29.80	.2645	.0005	......	13.14	.6010	......	.0009	29.73
2. Official 2½ per cent H_2SO_4 and 2½ per cent NaOH.	.5720	.0070	......	28.20	.2570	......	.0009	12.78	.5510	.0015	......	27.43
	.5765	......	.0009	28.43	.2495	.0005	......	12.40	.5540	......	.0009	27.58
3. 2½ per cent H_2SO_4 and 2½ per cent NaOH, but material not extracted with ether before treatment.	.6167	.0090	......	30.38	.2570	.0025	......	12.72	.5545	.0070	......	27.37
	.6184	.0200	......	29.85	.2490	.0025	......	12.32	.5550	.0065	......	27.42
	......	......	......	......	......	......	......	......	......	......	......	......
	......	......	......	......	......	......	......	......	......	......	......	......
	......	......	......	......	......	......	......	......	......	......	......	......
4. Hönig's method.	1.1100	.0410	......	51.25	.5040	.0135	......	22.15	1.8395	.0215	......	90.58
	1.1225	......	.0439	51.88	.4290	.0105	......	18.40	1.1220	......	.0191	54.16
	......	......	......	......	.4820	......	.0621	21.05	1.0720	.0180	......	51.66
	......	......	......	......	.6125	......	.0184	27.57	......	......	......	......
	......	......	......	......	.3855	......	......	16.22	......	......	......	......
	......	......	......	......	.4785	.0385	......	20.87	......	......	......	......
5. 1¼ per cent H_2SO_4	1.2150	......	......	......	.5460	......	......	......	1.1155	......	......	......
	1.2520	......	......	......	.5680	......	......	......	1.1370	......	......	......
	1.2220	......	.0662	......	.5200	......	.0402	......	1.0570	......	.0316	......
6. 1¼ per cent H_2SO_4 followed by glycerol, etc., as in Hönig's method.	.8210	.0435	......	37.52	.3880	......	.0094	18.88	.8155	.0075	......	39.50
	.8270	......	.0271	37.82	.3730	.0010	......	18.13	.8245	......	.0179	39.95
7. 1¼ per cent of H_2SO_4 and 4 per cent borax.	.8600	......	.0315	40.87	.3640	.0040	......	16.86	.8335	......	.0118	40.63
	.8560	.0110	......	40.67	.3694	......	.0228	17.13	.8265	.0090	......	40.28
8. 1¼ per cent of H_2SO_4 and silicate of soda	.7910	.0500	......	36.56	.3344	......	.0052	14.86	.7410	......	.0446	33.37
	.8050	......	.0097	37.26	.3210	.0320	......	14.19	.7255	.0290	......	32.59
9. 1¼ per cent H_2SO_4 and carbonate of soda.	......	......	......	......	......	......	......	......	......	......	......	......
10. 1¼ per cent H_2SO_4 and bicarbonate of soda.	......	......	......	......	......	......	......	......	......	......	......	......
11. Borax 4 per cent, 1 hour.	1.2040	.0030	......	56.33	1.1440	......	.0608	52.71	......	......	......	......
	1.2390	......	.0743	58.08	1.1235	.0290	......	51.68	......	......	......	......

TABLE I.—*Results of various solvents on feeding stuffs sent out by association reporters—Continued.*

Method used.	Corn meal.				Oil meal.				Clover hay.			
	Total resi-due.	Ash.	Albu-mi-noids.	Fi-ber.	Total resi-due.	Ash.	Albu-mi-noids.	Fi-ber.	Total resi-due.	Ash.	Albu-mi-noids.	Fi-ber.
	Gms.	Gms.	Gms.	Pr.ct.	Gms.	Gms.	Gms.	Pr.ct.	Gms.	Gms.	Gms.	Pr.ct.
1. Official 1¼ per cent H₂ SO₄ and 1¼ per cent NaOH.	.0425	......	.0009	2.03	.1710	......	.0009	8.23	.6410	(.0090)	.0087	30.05
	.0370	.0010	......	1.75	.1700	.0055	......	8.18	.6430	(.0090)	.0131	30.15
2. Official 2½ per cent H₂ SO₄ and 2½ per cent NaOH.	.0350	.0005	......	1.68	.1615	.0015	......	8.10	......	......	......	......
	.0370	......	.0008	1.78	.1545	......	.0009	7.60	......	......	......	......
3. 2½ per cent H₂SO₄ and 2½ per cent NaOH, but material not ex-tracted with ether before treatment.	.0405	.0030	......	1.87	.1790	.0060	......	8.65	......	......	......	......
	.0455	.0055	......	2.00	.1780	.0060	......	8.60	......	......	......	......
4. Hönig's method	.1515	......	.0183	6.31	.5085	......	.0704	18.63	.8295	.0270	......	38.33
	.2335	......	.0071	10.36	.5055	.0655	......	18.48	.8360	......	.0058	38.66
	.1625	.0125	......	6.86	......	......	......	......	......	......	......	......
5. 1¼ per cent H₂SO₄	.2285	......	......	......	.5125	......	......	......	1.0500	......	......	......
	.2350	......	......	......	.5025	......	......	......	1.0540	......	......	......
	.2250	......	.1096	......	.5300	......	.0882	......	1.0620	......	.0827	......
6. 1¼ per cent H₂SO₄ fol-lowed by glycerol, etc., as in Hönig's method.	.0615	......	.0009	2.98	.3015	......	......	13.51	.7485	.0045	......	35.97
	.0550	.0010	......	2.65	.3040	......	.0312	13.64	.8145	......	.0246	39.27
7. 1¼ per cent of H₂SO₄ and 4 per cent bo-rax.	.1200	.0030	......	3.35	.3090	.0145	......	13.98	.8745	(.0100)	.0875	38.85
	.1205	......	.0499	3.38	.3120	......	.0148	14.13	.8463	(.0100)	.0437	39.63
8. 1¼ per cent H₂SO₄ and silicate of soda......	.0780	.0035	......	2.80	.2770	.0220	......	11.61	1.2820	(.0500)	.0437	59.41
	.0805	......	.0184	2.93	.2780	......	.0218	11.66	1.1925	(.0500)	.0393	55.16
9. 1¼ per cent H₂SO₄ and carbonate of soda.	.0610	.0075	......	2.19	.2410	......	.0181	10.92	.7485	......	.0202	35.66
	.0639	......	.0096	2.34	.2305	.0145	......	10.39	.7430	.0130	......	35.49
10. 1¼ per cent H₂SO₄ and bicarbonate of soda.	.1160	......	.0530	2.82	.3240	......	.0508	12.93	.8550	......	.0486	39.27
	.1125	.0065	......	2.65	.3140	.0145	......	12.43	.8520	.0210	......	39.10
11. Borax 4 per cent 1 hour	......	......	......	......	......	......	......	......	.8290	......	.0387	......
	......	......	......	......	......	......	......	......	.8250	......	.0437	......

An examination of the table shows:

A.—That 2½ per cent acid and alkali gave lower results in every case than 1¼ per cent acid and alkali. [1–2].[1]

B.—That practically the same results are obtained on these samples, whether they are first extracted with ether or are directly treated with the acid and alkali [2–3]. The fiber obtained by treating with 2½ per cent acid and alkali without previous ex-traction with ether was lighter in color than that obtained by the use of acid and alkali of the same strength after treatment with ether. In case of corn bran, how-ever, the fiber obtained without extraction with ether contained fatty acids even after treatment with ammonia-cupric hydrate.

C.—That Hönig's method has not proved satisfactory in our hands. The differ-ence in duplicates is frequently very great, even where the conditions are kept as nearly uniform as possible. The amounts of albuminoids left in the fiber are too great to be neglected. In every case where pentosans were present in the original material they were found in the fiber obtained by Hönig's method from that mate-

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[1] Numbers in brackets refer to the numbering at left-hand side of Table I.

rial. In testing for pentosans the fiber was brought into a Kjeldahl flask, a small quantity of dilute sulphuric acid added, the flask connected with a short condenser, and a portion of the fluid distilled. The qualitative test for pentosans is made in this distillate. The flask is removed from the condenser, strong sulphuric acid and mercuric oxid added, and the nitrogen determined, one sample serving for both determinations. The amounts of fiber obtained by this method are much higher than that obtained by other methods. [4.]

D.—That the difficulties met with in using Hönig's method seemed to be due in great part to the presence of gums and starch. We therefore treated the feeding stuff with $1\frac{1}{2}$ per cent sulphuric acid before treating with glycerol. As the residues must be dried before treating with glycerol, it was thought best to also weigh them, and a nitrogen determination was also made in one of the residues from the acid treatment. [5]. The dry material was treated as in Hönig's method. The results are much lower than by the use of Hönig's method alone; the amount of albuminoids in the fiber is much lower; the filtering and washing of the fiber are easily accomplished, and this modification of the method seems to remove many of the difficulties connected with the original method. It seems better to apply the acid treatment before applying the glycerol, rather than after the glycerol treatment, as has been suggested, for much material is removed by the acid, and the glycerol is brought into more intimate contact with the fiber. [6].

E. That treatment with dilute sulphuric acid and 4 per cent borax solution gives a lower amount of fiber than Hönig's method, and less albuminoids are present in the residue. The albuminoids are however higher than in the residues from sulphuric acid and glycerol. The method is very easily carried out. [7].

F. Silicate of soda was tried in the place of caustic soda. It has replaced caustic soda in large machine shops for cleaning the cotton waste, since it does not render the fiber brittle. In order to keep the silica in solution it is made quite alkalin. The results obtained with it on feeding stuffs gave nearly the same results in fiber as borax or glycerol after preliminary treatment with dilute acid. The albuminoids are not completely removed. The method is as easy of application as the official method. [8].

G. Carbonate and bicarbonate of soda were tried in place of caustic soda on corn meal, oil meal, and clover hay. The bicarbonate foams badly and gives results high in both fiber and albuminoids. The carbonate works more satisfactorily, but it does not completely extract albuminoids, although on oil meal and clover hay it compares favorably with all other solvents except caustic soda. [9–10].

H. Borax alone was tried but the results were too high in albuminoids. [11].

I. Silicate of soda was also tried on the clover hay without previous treatment with acid. The results were lower in both fiber and albuminoids than on treatment with borax alone.

J. Doubling the strength of the solutions of silicate of soda and of borax had no effect in reducing the amount of fiber or albuminoids.

Throughout this section of the work the whole of the fiber from one determination was used for the determination of the ash and the whole of the fiber from another for the determination of nitrogen. While of course there are theoretical objections to this proceeding, I believe it involves less risk of error than would arise if we undertook to divide these residues and take a part of each for each determination of ash and nitrogen. It also has the advantage of reducing the manipulation considerably.

RESULTS ON SCHLEICHER & SCHÜLL FILTER PAPER.

This material was selected as a highly resistent cellulose. The work upon it included:

A. Influence of kind of solvent used.

B. Influence of strength of solution.

C. Influence of time of digestion.

D. Influence of combining the action of two solutions on the same sample of paper, the material being dried before the second solution was applied unless otherwise stated.

E. Influence of the oxygen in the air blast used.

The details and results appear in the following table:

TABLE II.—*Experiments with solvents on Schleicher & Schüll filter paper No. 589.*

[500 gram used containing water .0197 gram, ash, .0005 gram, and cellulose .4798 gram.]

Treatment with solvents.	Cellulose taken.	Cellulose recovered.	Loss of cellulose.	Loss.
	Gram.	*Gram.*	*Gram.*	*Pr. cent.*
Boiled thirty minutes with 2½ per cent H₂SO₄ 200 cc	.4798	.4715	.0083	1.73
	.4798	.4720	.0078	1.62
Boiled thirty minutes with 2½ per cent NaOH, 200 cc	.4798	.4385	.0413	8.83
	.4798	.4410	.0388	8.09
Boiled thirty minutes with 1¼ per cent NaOH, 200 cc	.4798	.4415	.0383	7.98
	.4798	.4395	.0403	8.39
	.4798	.4380	.0418	8.71
Boiled thirty minutes with water, 200 cc	.4798	.4747	.0051	1.06
	.4798	.4735	.0063	1.31
Heated to 210° C. with glycerol for thirty minutes, 60 cc	.4798	.4750	.0048	1.04
	.4798	.4730	.0068	1.42
Boiled thirty minutes with 4 per cent solution of borax, 200 cc	.4798	.4692	.0106	2.21
	.4798	.4700	.0098	2.04
Boiled thirty minutes with solution of silicate of soda, 200 cc	.4798	.4292	.0506	10.55
	.4798	.4315	.0483	10.07
Boiled one hour with 2½ per cent H₂SO₄, 200 cc	.4798	.4715	.0083	1.73
	.4798	.4730	.0068	1.42
Boiled one hour with 2½ per cent NaOH, 200 cc	.4798	.4228	.0570	11.88
	.4798	.4222	.0576	12.00
Boiled one hour with 1¼ per cent NaOH, 200 cc	.4798	.4330	.0468	9.75
	.4798	.4325	.0473	9.86
Boiled one hour with water, 200 cc	.4798	.4744	.0054	1.12
	.4798	.4732	.0066	1.37
Heated with glycerol to 210° C. for one hour, 60 cc	.4798	.4700	.0098	2.04
	.4798	.4670	.0120	2.40
Boiled one hour with 4 per cent solution of borax, 200 cc	.4798	.4727	.0071	1.48
	.4798	.4695	.0103	2.14
Boiled one hour with silicate of soda solution, 200 cc. Material in flasks gelatinized.				
Official method for fiber, 2½ per cent H₂SO₄ and 2½ per cent NaOH, 200 c. c., thirty minutes (residue after treatment with acid was dried and weighed)	.4798	.3750	.1048	21.84
	.4798	.3755	.1033	21.53
Official method, 2½ per cent H₂SO₄ and 2½ per cent NaOH, 200 cc., thirty minutes (residue after treatment with acid not dried)	.4798	.4000	.0798	16.68
Official method 2½ per cent H₂SO₄ and 2½ NaOH, but current of hydrogen used instead of air	.4798	.3508	.1290	26.94
Boiled with water one-half hour and 2½ per cent NaOH, ½ hour, 200 cc. each	.4798	.4370	.0428	8.92
Boiled with 200 c. c. 2½ per cent H₂SO₄ one-half hour and then treated with 60 cc. glycerol at 210° C. one-half hour	.4798	.4650	.0148	3.08
Boiled with 2½ per cent HCl one-half hour, 200 cc	.4798	.4655	.0143	2.98
	.4798	.4670	.0128	2.66
Boiled with 2½ per cent HCl and 2½ per cent NaOH one-half hour, 200 cc. each	.4798	.3065	.1733	36.12
	.4798	.3060	.1738	36.22

Generally stated the results are:

A. Water, glycerol, borax, hydrochloric acid 2½ per cent, and sulphuric acid 2½ per cent have comparatively little solvent action on the paper. Caustic soda and silicate of soda dissolve considerable quantities of the paper.

B. Increasing the strength of the caustic soda solution causes more loss of fiber, although the increase is not great.

C. With sulphuric acid, water, and borax, an additional boiling for 30 minutes gave no increase in amount of paper dissolved. With caustic soda and glycerol, a marked increase in solvent action results from increased time of digestion. With silicate of soda, the material gelatinized before the one-hour digestion was completed. As the alkalin silicate of soda attacks the fiber vigorously, and as the product of treating cellulose with alkali is an acid, this gelatinization was probably due to separation of silicic acid.

D. With a single exception (water followed by caustic soda), more fiber was destroyed when two solvents acted one after another on the same sample of pulp, than the sum of the fiber destroyed by the same solvents acting on two separate portions of the pulp. This is very noticeable in the process used as an official method for fiber, where the increase is from 10.13 per cent with separate samples of pulp to 16.68 per cent where the two solvents acted on the same sample of pulp; and when hydrochloric acid was used in place of sulphuric, an increase was from 11.28 per cent on separate samples to 36.17 per cent when the reagents acted on the same sample of pulp.

E. As cellulose is said to be readily oxidized by boiling with even dilute alkali in the presence of oxygen, and as the air blast has been used to prevent foaming in the above work, a stream of hydrogen was substituted for the air blast. The result was rather unexpected, as when the hydrogen was used a loss of 26.94 per cent occurred, against a loss of 16.68 per cent with air.

EXAMINATION OF FIBER OBTAINED BY TREATING FEEDING STUFFS BY THE OFFICIAL
METHOD AND WITH DILUTE SULPHURIC ACID AND GLYCEROL.

This work is but just begun and the only results now ready are those on the sample of timothy hay, and these results are but partial. The results so far obtained appear in the following table:

TABLE III.—*Experiments with solvents on fibers.*

Reagents.	Appli-cation.	Fiber from timothy hay by glycerol and H_2SO_4; per cent dissolved.		Fiber from timothy hay by official method; per cent dissolved.	
		A	B	A	B
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Schweitzer's ammonic cupric hydrate	First	21.26	21.93	16.30	7.50
Chlorin water	... do	11.91	14.29	17.40	16.50
Nitric acid and chlorate of potash	... do	5.47	6.38	4.56	4.48
Schweitzer's ammonic cupric hydrate	Second	11.91	12.90	15.34	14.75
Chlorin water	... do	1.68	2.64	14.12	12.80
Nitric acid and chlorate of potash	... do	2.70	2.35	....	....

The most conspicuous feature of this table is the marked difference in the amount of material removed from the two fibers by the second treatment with chlorin water. Not much more than one-half of the fiber has been dissolved. What remains has the appearance of small sticks of wood. No doubt the fineness of the grinding will have a marked influence in work of this character. The materials will be examined further.

The determination of fiber in feeding stuffs is said to be a rough imitation of the process of digestion in the animal. Our present methods seem rather to be very crude attempts at paper making. If we are to imitate digestion processes why are the fats first removed? Why should sulphuric acid be substituted for hydrochloric? Why should solution at boiling temperature replace the action of ferments? Are we justified in putting the one as the equivalent of the other even in relatively rough comparisons?

While it is admitted that sweeping changes in methods are undesirable since they render less useful past work, there seems to be a strong necessity for some work of a different character on the more insoluble constituents of feeding stuffs before we can accept the analysis of a cattle food as furnishing any very reliable evidence of its feeding value.

The two recommendations of the reporter on cattle foods were again read. On motion, the first was adopted. The question was then upon the adoption of the second.

Mr. TELLER moved to amend by directing that the hydrogen be passed through the material.

Mr. WOLL. Then it will be necessary to specify the time, and I think that under those conditions five hours will prove ample. If Mr. Teller will include the five-hour limit I will be satisfied.

Mr. HUSTON. I would make it read not less than five hours. There are some cases where it may be desirable to extend the drying a little further.

The recommendation, as amended, was adopted.

It was moved that the adjournment, when taken, be until immediately after the address by Dr. George Lunge before the Chemical Congress on the following morning.

The motion was carried.

Mr. VAN SLYKE moved that the methods adopted by the association be regarded as official and in effect upon the publication of the bulletin.

After some discussion, Mr. Frear suggested, as an amendment, that the methods, so far as they applied to fertilizers, should become official on January 1. The amendment was accepted, and the motion, as amended, was carried.

At 5:30 p. m. the convention adjourned.

THIRD DAY.

MORNING SESSION, SATURDAY.

The convention was called to order by the president at 11:30 o'clock, who then asked for the report on fermented liquors.

Mr. Crampton read the following paper:

REPORT ON FERMENTED LIQUORS.

By C. A. CRAMPTON.

The work upon this subject was carried out on the same general plan as last year, a sample of dry, and one of sweet, wine being sent out to those signifying a desire to take part, accompanied by the following letter of instructions:

TREASURY DEPARTMENT,
OFFICE OF THE COMMISSIONER OF INTERNAL REVENUE,
Washington, D. C., March 21, 1893.

DEAR SIR: I send you to-day by express a box containing two samples of wine. I should be glad to have you determine alcohol and extract in these samples, by association methods, as prescribed on pages 232 and 233, Bulletin 35.

If you have the time I should also like to have comparisons made of extract determinations by the following method.

Weigh out from a stoppered flask or weighing bottle 5 grams of the dry wine into a flat bottomed platinum dish, about 50 mm. in diameter, 10 mm. high, and 25 cc. in capacity. Run the wine evenly over the bottom of the dish, place directly in a drying oven, and dry for three hours at a temperature of 100°C. Of the sweet wine 2 grams only should be weighed for the determination.

A comparison and discussion of the proper way to determine and express the percentage of alcohol by volume, especially in sweet wines, would be very desirable. It has been suggested to me by a member of the association that an easy method of making a comparison between the percentage of alcohol by volume, as obtained by measuring the volume of the sample and distillate, taking the sp. gr. of the latter, and finding the percentage by volume from the tables, and the result as obtained by calculation from the percentage by weight, would be by measuring the volume of the sample and distillate, and weighing them as well. In this way a single operation will serve for both calculations. The data for these calculations should be reported in full.

The work should be done as soon as possible after the receipt of the samples, the latter being kept lying on their sides in a cool place. Results sent in later than July 1 will not be included in the report.

Respectfully yours,

C. A. CRAMPTON,
Reporter on Fermented Liquors

The results were somewhat disappointing, but only in respect to the small number of reports received. This was due to various causes. One set of samples was broken in transit; one analyst was taken ill and could not do the work; one reported that he was obliged to give it up on account of pressure of other matters; two requests for samples were received too late, and four of those who received the samples sent in neither reports nor explanations. So that of the sixteen sets of samples sent out only nine were analyzed and reported upon.

The following table gives the results obtained on alcohol by weight and total solids:

Analyst.	Riesling.		Angelica.	
	Alcohol by weight.	Total solids.	Alcohol by weight.	Total solids.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
J. A. Heberly (Arizona)	9.18	1.90	14.41	15.00
G. L. Teller (Arkansas)	9.76	2.13	14.43	15.59
George E. Colby (California)	9.45	2.11	14.58	15.60
Charles Ryan (Colorado)	[1]9.55	[1]1.90	[1]14.27	[1]13.14
H. A. Huston (Indiana)	9.78	2.12	14.59	15.77
H. J. Patterson (Maryland)	10.27	2.08	15.13	15.52
F. S. Shiver (South Carolina)	9.71	2.12	14.44	15.55
W. H. Krug and J. S. Carman (U. S. Department of Agriculture)	[2]6.83	1.65	[2]10.97	14.95
C. A. Crampton (U. S. Internal Revenue)	9.84	2.09	14.59	15.58
General average	9.70	2.01	14.55	15.19
Difference between highest and lowest	1.09	.48	.86	2.63

[1] See accompanying letter.
[2] These results omitted from the averages.

The results show a very marked improvement over the work of last year. The only report which showed a serious variation from the average was that of the Department of Agriculture. It was explained from the fact that the samples were allowed to stand several months in the laboratory before analysis, without refrigeration. It does not seem likely that the fortified wine would change, even under these conditions, but this seems the only explanation. The results are excluded from the general average.

The method of procedure and data for calculation are given by most of the analysts in a very complete and satisfactory form. Some of the notes and comments made are appended.

Arkansas.—The bottles were opened April 10 and 11, about two weeks after they were received. In the determination of the per cents of alcohol the method of procedure was essentially the same as that of last year. The wine and distillate were both measured and weighed in the same flask and their volumes are assumed to be equal, though variations in temperature might have caused a slight difference.

In determining the solid extract, a glass dish 5 cm. in diameter and 3 cm. deep, with straight sides and a flat bottom and having a capacity of 60 cc. to the brim, was used instead of the platinum dish as directed. This dish was chosen to allow the using of a form of drying bath of which a sketch is inclosed that will, I think, explain itself. This form of bath was devised to give the full temperature of boiling water in drying the ether extract from fodders and has been used in this laboratory for about two years with quite satisfactory results. Its advantage lies in that the actual temperature of boiling water is maintained in the drying-chamber, a condition which I have been unable to obtain either with the usual form of water oven or the air bath with thermostat connection. The temperature of the air in this bath will be usually not more than a fraction of 1 degree below the actual boiling point of the water. The air may be replaced with hydrogen by passing it in through the tubular in the cover. Have found the hydrogen escaping from an exit tube passing through the same tubular to burn quietly and steadily for hours though it is not usually used in my work. The flasks and dishes are introduced into the chamber by means of a pair of crucible tongs, and though this is somewhat inconvenient, it seems much more than compensated for by the satisfaction of having a definite temperature. Have found

the glass dishes to work very satisfactorily for the determination of the extract. They are cheap, neat, and easy to handle.

California.—Acidity of Riesling, 0.637 per cent; as tartaric of the angelica, 0.337 per cent (after iv p. 233, Bull. 35). The angelica showed 14.15 per cent reducible sugars; after inversion the sugar amounted to 14.22 per centum (after vii p. 233, Bull. 35).

Colorado.—Three determinations were made on the same sample and the per cent of alcohol obtained from tables given in Bulletin 35. without making corrections for difference in temperature. All determinations were made at 23° C. Owing to the high elevation (4.996 feet) the boiling point of water is only 95° C., and we would expect the per cent of alcohol distilled over to be less than that distilled at a lower elevation. [? ED.]

Indiana.—All specific gravities are with pycnometer whose content and thermometer had been carefully checked up. In the determination of extract the time of drying was strictly followed.

Maryland.—All weighings and measurements were made at or near 15.5° C. All dryings were made in a water-oven which gives a temperature of 98° to 99° C. The sp. gr. of the distillates were obtained by the Westphal balance. The extract by the indirect, alternate, method was obtained by using the sp. gr. as obtained by the Westphal balance. Work prepared on each 17 and 18.

All the analysts except one made comparisons of the results obtained by film drying with those given by the official methods, as requested in the letter of instructions. Following is the table of comparative results obtained:

	Riesling.		Angelica.	
	Associa-tion.	Film drying.	Associa-tion.	Film drying.
	Per cent.	Per cent.	Per cent.	Per cent.
Teller	2.13	2.13	15.59	15.95
Colby......................	2.11	2.12	15.60	15.37
Huston	2.12	1.88	15.77	15.45
Patterson....................	2.08	1.70	15.52	15.36
Shiver......................	2.12	2.00	15.55	15.61
Krug & Carman	1.65	1.71	14.95	15.00
Crampton	2.09	1.63	15.58	14.98
Average..............	2.04	1.88	15.51	15.39

The average shows a minus difference of 0.16 in the case of the dry wine, and 0.12 in the case of the sweet wine. While these figures are satisfactory, I do not know that I am prepared to recommend the adoption of the film-drying method in lieu of the present official method, at least not without another season's trial. I am inclined to think that the time recommended by me for drying in the film-method is too long, and that two hours' heating would be sufficient. It might be well to prescribe the longer period for the sweet wines and the shorter for dry wines. The method of drying in hydrogen for wine residues might profitably be studied another season also.

The suggestion as to a comparison of the percentage of alcohol by volume as obtained directly from volume measurements and as calculated from the determination by weight was followed by only two or three analysts, and perhaps was not clear enough to be well understood. The most complete report and discussion upon this point was made by the South Carolina Station, and is so important that I append it in full:

ALCOHOL.

"Measured wine in a 100 cc. flask at 15.5° C and weighed. Transferred to a 300 cc. distilling flask, washed out weighing flask with 50 cc. of distilled water and

added washings to distilling flask. Distilled into the original 100 cc. measuring flask until distillate nearly reached mark, made up with distilled water to mark at 15.5° C and weighed. The specific gravity was then taken by pycnometer at 15.5° C. As the measuring flask was not specially calibrated, the specific gravities taken with pycnometer were used in all the calculations instead of the figures given by $D = \dfrac{W}{V}$.

Although the work was so arranged in each analysis that a single distillation furnished the necessary data for comparing the per cent of alcohol by volume in the wine as taken from table with that obtained by calculation from the directly determined percentage by weight, the results are, for the sake of clearness, arranged as if separate experiments had been made by weight and by measure.

"Method I.—*By weight.*

	Riesling.		Angelica.	
	First.	Second.	First.	Second.
Sp. gr. of wine at 15.5° C. (pycnometer)	.99241	(.99241)	1.0408	(1.0408)
Sp. gr. of distillate at 15.5° C. (pycnometer)	.98419	.98408	.97716	.97716
Weight of wine taken (grams)...........................	99.2080	99.2428	104.0436	104.0088
Weight of distillate (grams)	98.3705	98.3785	97.7877	97.7481
Per cent of abs. alcohol by weight in distillate (table)	9.79	9.87	15.36	15.37
(1) Per cent of abs. alcohol by weight in wine..............	9.71	9.78	14.44	14.44
(2) Per cent of abs. alcohol by volume in wine.............	12.14	12.23	18.93	18.93

(1) Weight of distillate × per cent of abs. alcohol by weight in distillate (table) ÷ weight of wine taken.

(2) $V = a\dfrac{S}{s}$: in which

$a =$ per cent by weight of alcohol in the wine.

$S =$ sp. gr. of wine.

$s =$ sp. gr. of alcohol 0.7939.

"Method II.—*By measure.*

	Riesling		Angelica.	
	First.	Second.	First.	Second.
Sp. gr. of wine at 15.5° C. (pycnometer)	.99241	(.99241)	1.0408	(1.0408)
Sp. gr. of distillate at 15.5° C. (pycnometer)	.98419	.98408	.97717	.97716
Volume of wine taken cc. (flask)........................	100	100	100	100
Volume of distillate cc. (flask).........................	100	100	100	100
Per cent of abs. alcohol by vol. on distillate (table)........	12.15	12.25	18.91	18.92
Per cent of abs. alcohol by vol. on wine	12.15	12.25	18.91	18.92
Per cent of abs. alcohol by weight on distillate (table)....	9.79	9.87	15.36	15.37
[1] Per cent of abs. alcohol by weight on distillate wine.....	9.71	9.79	14.42	14.43

[1] Sp. gr. of distillate × vol. of distillate × per cent of abs. alcohol by weight in distillate ÷ sp. gr. of wine × vol. of wine.

"Comparing the results on volume per cents, we find:

	Riesling.		Angelica.	
	First.	Second.	First.	Second.
	Per cent.	Per cent.	Per cent.	Per cent.
Volume per cent calculated from weight	12.14	12.23	18.93	18.93
Volume per cent table...........................	12.15	12.25	18.91	18.92

"It thus appears that in a wine the percentage by volume of alcohol taken from table is practically the same as that calculated from the percentage by weight. This follows from the fact that the percentage by volume in the distillate is correct for the wine, provided the volume of the distillate is the same as that of the wine at the standard temperature. Of course it is understood that this "percentage by volume" does not represent the volume which the alcohol actually occupies in a hundred volumes of either distillate or wine, but the volume which it would occupy if separated from a hundred volumes of either by distillation.

"Even if the contracted volume of the alcohol in an alcoholic liquor could be accurately determined, the result would, for practical purposes, be undesirable and misleading, as it would be apt to convey the idea that the liquor contained less alcohol than really existed in it, and would, moreover, render it impossible to calculate in any case the correct percentage of alcohol by weight from the given percentage by volume."

It would seem from the above that the conclusions reached in the report quoted were correct, and that either of the methods for obtaining the percentage of alcohol by volume is accurate and the results correspond. Previous to this season's work I had thought differently, but I found the mistake I made was in taking the percentage of alcohol by volume directly from the table opposite the corresponding per cent by weight, which gave a much lower figure in the case of sweet wines than that obtained by using measured volumes and taking the percentage directly from the specific gravity of the distillate. Applying the same calculation as is made above to the data sent in we have the following:

ANGELICA.

	Alcohol by volume.	
	By calculation.	Direct.
	Per cent.	Per cent.
South Carolina..............	18.93	18.92
Arkansas...................	19.07	18.92
Indiana...................	19.11	19.11
Internal Revenue...........	19.12	19.13
Average..............	19.06	19.02

RIESLING.

	Alcohol by volume.	
	By calculation.	Direct.
	Per cent.	Per cent.
South Carolina..............	12.14	12.15
Arkansas...................	12.15	12.18
Indiana...................	12.22	12.23
Internal Revenue...........	12.30	12.30
Average..............	12.20	12.21

I would recommend the continuance of existing methods for another year without change.

On motion of Mr. Frear the recommendation was adopted.

The president called for the report of the abstracting committee.

Mr. Frear read the following report:

REPORT OF THE ABSTRACT COMMITTEE.

By Wm. Frear, *Chairman.*

Mr. President and Members of the Association: Your committee beg leave to make the following brief statement as introductory to the body of their report, which will not be read at this time.

The first duty devolving upon them has been the adoption of a system, including the form of the abstract, the number of data to be included, the style of abbreviation to be adopted, the scope to be assigned to the several classes of subject-matter dealt with, the final arrangement of the collection of abstracts, and the distribution of the labor of abstracting. These matters have all been carefully considered by your committee, since it is deemed of the highest importance for future reference that the method, once settled, should be carefully adhered to in succeeding years should it seem wise to continue the work.

As to the form of the individual abstract: This is to consist (1) of the name of the author; (2) of the date of first public presentation of the article when the date of printing is so remote from that of the paper as to make priority doubtful judging from date of issue alone; (3) a brief English title embodying the distinctive point involved; (4) the original title of the paper in the original language when this can be obtained; (5) the abstract proper; (6) the abbreviated title of the journal of original issue, with series, volume, and pages noted (the volume number indicated by bold-faced type in Arabic numerals rather than Roman); (7) the references to journals in which abstracts may be found; (8) the initials of the abstractor.

I may say here that there was a marked difference of opinion touching several points in the above order, but the above sequence is that favored by the majority of the committee.

As to the abbreviations of the titles of periodicals to which reference is made: It was decided to adhere to the recommendations of the committee on chemical bibliography of the Association for the Advancement of Science, so far as they go, and in case of journals not included in their lists to follow their principles in the adoption of an abbreviation.

In selection of matter and giving it perspective the following order of importance was laid down: (1) Papers of major importance to methods of most direct agricultural application; (2) papers of minor importance in the same field; (3) papers of major importance in correlated branches; (4) papers of less importance in the same field. Such shaping of perspective must vary, however, very largely with the individual abstractor, and it is not to be expected that perfect uniformity in such shaping should be obtained by the collaboration of a number of abstractors.

It was further believed that the most satisfactory arrangement of the abstracts would consist (1) in their grouping under certain broad subject heads; (2) their subordinate arrangement alphabetically by author; (3) their numbering in their rough subject classification, and (4) a brief author index, arranged alphabetically, and giving in addition only a cross reference to the numerical position of the abstract of any paper written by any given author. The brief English title of the paper will suffice for a subordinate subject classification. Should the work be continued in succeeding years, any paper could be briefly referred to by indication of the year and abstract number.

It was decided also that the work for 1892–'93 should be limited, so far as original papers were concerned, to those published from July, 1892, through June, 1893, and

so far as abstracts appearing in chemical publications are concerned none representing papers originally published prior to January 1, 1892, should be included. So far as possible, these limitations have been observed.

It was furthermore found impossible, owing to the late day upon which the *modus operandi* was finally fixed, to cover the entire field of periodical literature. The work embodied in the accompanying report[1] consists, however, of abstracts and titles taken from 80 journals, representing 359 authors.

The numerical distribution of these abstracts among the several more important subjects is as follows: Phosphoric acid, 31; nitrogen, 30; potash, 15; soil and ash analysis, 15; feeding stuffs, 16; sugars, etc., 85; fermented liquors, 35; dairy products, 94; miscellaneous, 101.

The chairman of the committee desires to express his appreciation of the hearty coöperation given by his fellow members on the committee—Messrs. Allen, Ross, Winton, and Woll—given despite heavy pressure of local business and the unfortunate but unavoidable retarding of the beginning of the work proper; and also for the aid given by his assistant, Mr. J. N. Fields, in covering a large portion of the abstracting work assigned to himself. It is believed by your committee that this system, if properly sustained, will result in the building up of the most valuable "Jahresberichte" of agricultural chemical analyses to be found in the English language.

The report was adopted.

The report on sugar analyses was next called for.

In the absence of Mr. Spencer, Mr. Huston read the following paper:

REPORT ON SUGAR.

By G. L. Spencer.

The association instructed the reporter on sugar to investigate the following methods: Drying in vacuum and in air; determination of water from density (Pellet's method); determination of ash by method of Alberti and Hempel; determination of reducing sugars by gravimetric method according to Formanek as modified by B. B. Ross.

In sending out samples for use in investigating these methods the reporter also requested the estimation of sucrose by the method of Clerget, heating ten minutes, and by the official German method, heating fifteen minutes. But four chemists, aside from those of the U. S. Department of Agriculture, reported on the samples sent them.

As stated by last year's reporter, it is difficult to select samples for this work which are not very liable to alteration. One of the samples (No. 1) prepared this year did not prove free from this objection. Sample No. 1 was a molasses from "first sugar" (unwashed) and No. 2 a molasses from "second sugar." In order to secure uniform samples, the molasses in each case was diluted with boiling water, then concentrated in a small vacuum pan and run directly into the bottles.

The only alteration reported was in sample No. 1 (Messrs. Crawley, Fuelling, and Carman) in which the sugar had crystallized on the sides of the bottle. The samples were very dark, rendering polarization of dilute solutions necessary and of course increasing the probability of error.

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[1] The report was received too late for insertion in this bulletin.

The results obtained by the various analysts are presented in the following table:

	Ash.			Solids.				
	Ordinary method.	Alberti & Hempel.	Sulphate.	Open dish (Official).	Vacuum.	Dilution and specific gravity.	Pellet's method.	Josse's method.
Sample No. 1.	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
F. S. Shiver, Clemson Agricultural College	....	5.91	....	79.76	80.22	....	....	....
J. T. Crawley, Louisiana Sugar Experiment station..............	6.63	6.00	8.83[1]	....	....	....	....	....
B. B. Ross, Louisiana State Experiment station.....................	5.81	....	....	77.25	76.13	....	....	....
R. E. Blouin, Louisiana State Experiment station..................	5.80	5.29	....	77.39	76.39	82.99	78.00	....
E. E. Ewell, United States Department of Agriculture.............	....	....	....	....	....	85.03	....	....
J. S. Carman, United States Department of Agriculture.........	6.62	6.09	8.03[2]	{76.66[3] / 78.37}	75.47	....	....	73.69
J. L. Fuelling, United States Department of Agriculture.........	....	....	....	....	....	....	83.67	....
Sample No. 2.								
F. S. Shiver, Clemson Agricultural college......................	....	6.39	....	78.57	78.96	....	....	....
B. B. Ross, Louisiana Sugar Experiment station.................	....	6.44	....	77.45	76.16	....	....	....
R. E. Blouin, Louisiana State Experiment station..................	7.12	6.33	....	77.81	75.83	83.38	78.30	....
E. E. Ewell, United States Department of Agriculture	....	....	....	....	....	84.35	....	....
J. S. Carman, United States Department of Agriculture.........	7.49	6.28	8.40[2]	{76.11[3] / 75.86}	73.86	....	....	72.84
J. L. Fuelling, United States Department of Agriculture.........	....	....	....	....	....	....	82.68	....

[1] Reported as sulphated ash. No deduction.
[2] A deduction of $\frac{1}{10}$ made.
[3] Dried 35 hours.

	Reducing sugars.				Sucrose.		
	Volumetric.	Electrolytic[1] A.	Electrolytic[2] B.	Cu. reduced in H.	Single polarization.	Double polarization. Ten min.	Double polarization. Fifteen min.
Sample No. 1.	Per cent	Per cent	Per cent	Per cent	Per cent.	Per cent.	Per cent.
F. S. Shiver, Clemson Agricultural College	....	14.37	....	14.24	41.2	45.25	....
J. F. Crawley, Lousiana Sugar Experiment station....................	....	....	....	....	{ 39.4 / 39.5	45.5	46.5
B. B. Ross, Louisiana State Experiment station.....................	....	16.80	16.85	....	....	....	....
R. E. Blouin, Louisiana State Experiment station..................	16.80	16.81	16.93	....	43.2	46.22	46.16
E. E. Ewell, United States Department of Agriculture..............	....	16.91	16.86	....	{ 40.4 / 40.7	13 min. 45.2	44.1
J. S. Carman, United States Department of Agriculture..........	....	....	....	....	....	....	....
J. L. Fuelling, United States Department of Agriculture.........	....	....	....	....	....	....	....
Sample No. 2.							
F. S. Shiver, Clemson Agricultural College	....	24.52[3]	....	24.66	23.0	29.99	....
B. B. Ross, Louisiana State Experiment station	....	28.42	28.28	....	....	....	....
R. E. Blouin, Louisiana State Experiment station..................	28.10	28.16	28.14	....	27.5	34.09	33.94
E. E. Ewell, United States Department of Agriculture..............	....	28.91	28.87	....	22.1	13 min. 30.0	30.4
J. S. Carman, United States Department of Agriculture..........	....	....	....	....	....	....	....
J. L. Fuelling, United States Department of Agriculture.........	....	....	....	....	....	....	....

[1] Electrolytic A.=Formanek method as modified by B. B. Ross.
[2] Electrolytic B.=Alternate method, p. 208, Bul. 35.
[3] Subacetate of lead added, and after filtration, excess of lead removed by carbonate of soda.

ASH.

The important consideration in connection with the determination of the ash, especially in sugars, is the commercial one of "rendement." As is well known, the amount of ash is taken into account in the calculation of rendement by the refiners. It seems to the reporter that this should influence the association in adopting a method. The evidence available indicates that the results obtained by the Alberti-Hempel method, employing quartz sand, show very nearly the true amount of organic matter in the sample, the organic acids being replaced by silicic acids and inorganic salts remaining unchanged, with the exception of the nitrates. The usual method of incineration, especially with beet sugars, is that marked (d) under the official methods, except that it is customary to deduct one-tenth to correct for the conversion of the mineral matters into sulphates. This is the method usually employed in calculating rendement, and this is probably the only argument in its favor. If we are searching a method giving as nearly as possible the true amount of ash, that of Alberti and Hempel should be adopted. If we wish to comply with the usage of the refiners, Scheibler's method, using sulphuric acid, should be employed, at least for the present.

There are very considerable differences in the results reported on the carbonated ash in sample No. 1. The results obtained by Messrs. Ross and Blouin agree very closely with one another, but are considerably lower than those of Messrs. Crawley and Carman. The results obtained by all analysts by the Alberti-Hempel method agree very closely.

SOLIDS.

Judging from the results obtained, the estimation of the solids in sugar samples presents greater irregularities than any other work. The results obtained by the official method, drying in an open dish, are more uniform than others reported, but experience demonstrates that these figures show far from the true percentage of solid matter. For example: In an experiment made in the Department of Agriculture, samples continued to show a decided loss on drying, even after 165 hours. According to the official method it is stated that the drying must be continued until the loss in 1 hour is not greater than 0.2 per cent. Even after this point was reached samples continued to lose for more than 140 hours, the total additional loss amounting to as much as 7 points. Such results are certainly far from satisfactory.

In the case of drying in vacuum it was also noticed that the samples continued to lose water after many hours.

By Pellet's method, that is, by the determination of the specific gravity of a solution containing a definite amount of the sample and reference to a table, results were obtained showing considerable variations. Of course, in work with samples of molasses from cane sugars, it would be necessary to prepare a special table. With such a special table, compiled from actual dryings of samples of molasses, it is undoubtedly practicable to obtain fairly good results by this method.

Methods employing dilution, the taking of the specific gravity, and calculation, should be rejected, since, owing to the accumulation of mineral matters in low products, the percentages of solids indicated will be far from correct. For sugar-house purposes, however, such methods might be adopted, employing arbitrary tables based on preliminary analyses, which would give results for use in comparative work.

Long-continued drying of samples in vacuum gave considerably lower percentages of solids than were obtained by the open-dish method. In vacuum drying it required practically more than 50 hours to obtain a fair estimate of the solids.

In addition to the methods cited, experiments were made with that of Josse (See Bulletin de l'Association des Chimistes, 10th year, No. 9, p. 656). This method consists essentially in dissolving a portion of the sample in water and absorbing the solution upon a coil of filter paper, upon which it is finally dried. The drying is conducted at a temperature between 100° and 110°. Estimations made by this method indicated that in about 12 hours the drying is much more complete than even after more than 100 hours in vacuum. The results obtained on the samples by this method are given in the table.

REDUCING SUGARS.

The results of the estimation of reducing sugars by the official electrolytic method and that of Formanek are very uniform, with the exception of the results obtained at the Clemson Agricultural College. In this laboratory in one set of analyses the copper was reduced in hydrogen. The results obtained by the two methods agree very closely with one another. The materially lower percentage of reducing sugars obtained was probably due to failure to acidulate the solutions before filtering off the subacetate of lead precipitate. Owing to the failure to add acetic acid a portion of the reducing substance was undoubtedly carried down with the lead precipitate and was filtered off.

The modified method of Formanek is simpler than the methods adopted by this association last year and the results obtained seem to be perfectly satisfactory.

SUCROSE.

The results of the methods of sucrose estimation employed by the different analysts were not reported in sufficient detail to enable a study of the work. The variations in the direct polarization are considerable. Under the circumstances, the results obtained do not justify any suggestions for the modification of the methods.

ABSTRACTS OF NOTES BY ANALYSTS.

Laboratory, Clemson Agricultural College.—In the estimation of the sucrose the use of acetic acid was omitted.

Laboratory, Louisiana Sugar Experiment Station.—Sample No. 1 only was examined. The material had suffered no decomposition from fermentation or any other cause that could be detected. The sugar had, however, crystallized on the sides of the containing vessel. It did not thoroughly dissolve even on allowing the bottle to stand in boiling water for one-half hour. The incineration with sand proceeded quietly and without the expenditure of a great deal of time. The duplicates agreed well and the method seems a favorable one.

Laboratory, Louisiana State Experiment Station.—B. B. ROSS: "I give, first, the proportion of solids as determined by dilution, taking specific gravity and then referring to a table for the degrees Brix and making calculation as usual. Of course with low-grade products, the apparent proportion of solids is far above the actual content as shown by drying.

"The proportion of solids, by dilution, taking density and referring to Pellet's table for molasses (beet), is within less than a unit, in each case, of the figures obtained by determining solids in the water oven. As this table of Pellet's applies to molasses of a somewhat higher degree of purity, a still smaller difference would have been noted, had a table for low-grade molasses been used.

"To secure even approximately constant weights in water oven, a drying for forty hours was, in some cases, necessary, and even in vacuum a slight loss in weight could be secured by continuing the drying beyond twenty-four to thirty hours.

"In determining reducing sugars, in addition to the volumetric estimations, comparative tests were made of the method originally published in the seventh annual proceedings, and the modified method of Formanek. The first method provides for the solution of Cu_2O in hot dilute nitric acid, and evaporation nearly to dryness after addition of H_2SO_4. The latter process was executed in the manner described in the February number of the Journal of Analytical and Applied Chemistry, without attempting to dissolve the Cu_2O on filter, but replacing asbestos, precipitate and all, in beaker used in precipitation, adding 4 per cent HNO_3 solution and electrolyzing in usual manner, employing as the anode a flat spiral of platinum, which rests on the bottom of the beaker, under the asbestos. This better insures the solution of the Cu_2O.

"In ash determinations, I found that the Alberti-Hempel quartz sand method gave extremely quick ash estimations, stirring the mass not even being necessary in many cases and scarcely a trace of char being observable in residue."

Laboratory, U. S. Department of Agriculture.—Messrs. Carman and Fuelling report crystallization of sugar in sample No. 1. Extensive experiments were made with the vacuum method of drying. It was observed that a considerable amount of the material containing reducing sugar was carried over with the distillate, even in small quantities when special precautions were taken. This was probably due to the poor quality (from a sugar-making point of view) of the material. It is often difficult in sugar-house practice to prevent loss in the evaporation of low products in vacuum.

In the work reported by this laboratory, a preliminary drying was made in a slow current of air, and afterwards in the case of No. 1, 35 hours, and No. 2, 49 hours, in vacuum. There was no loss by entrainment. The drying in each case was continued as long as there was a decided loss.

The following changes in methods are recommended by the reporter:

On page 204, Bulletin 35, under "determination of water," second line, substitute "ten" for "three."

Under "determination of ash" page 205, (*d*) insert a factor to be employed in the calculation of results. The factor 0.9 is recommended not on account of giving reasonably accurate results, but since it is usually employed in correcting sulphated ash for use in calculating rendement.

Page 205, after (*e*) insert the method of Alberti and Hempel for ash determinations. (See Bulletin 35, p. 162.)

The alternate method for determining the weight of copper in reducing sugar estimations as given on page 208 and the modified method of Formanek (p. 212) are certainly as accurate as if not more so than the official gravimetric method (p. 207), and should be made official.

I further recommend that the reporter for next year be requested to investigate drying in air, in vacuum, and by Josse method.

Mr. Ross, referring to the method of Alberti and Hempel for determination of ash, said that the character of the quartz sand flour should be distinctly specified. Some that he had recently purchased was of very inferior quality, and he could do nothing with it. He had therefore prepared some of his own from pure quartz crystals, and then obtained good results.

Mr. Huston said that if one used any considerable quantity of quartz sand he might find it profitable to correspond with manufacturers of sandpaper, who used an excellent quality in making the finer grades.

Mr. Ross. I would like to say with regard to that method of Josse which is recommended, it seems to me we have essentially the same method stated among our alternate methods, or at least it has been among them until very recently. Of course the method should be tested, but I think that it is already one of our official or provisional methods. I think this method proposed by Josse as something new is essentially the same thing proposed and used by Dr. Wiley some years since.

Mr. Wiley considered the first recommendation the most important. As to water determination, he thought that to say he was all at sea would be to use a very proper expression. The more he worked upon it the less he knew about it. Either the work should be stopped and some primitive method resorted to, or else, if possible, some method should be found by which the actual amount of solid matter in the viscous substances which came to the manufacturer, sugar maker, and chemist—the sirups, low grade molasses, and everything of that kind—could be determined. A paper he had hoped to be able to prepare in time for presentation at this time contained some very remarkable data in this regard. By the continued drying of a sample of honey, which by the official method would yield about 17 or 18 per cent of water, he had, after drying in vacuum, on paper coils, on paper slips, on kieselguhr, and in all the various ways, after solution in alcohol and absorption in the same, and so on, got over 40 per cent of volatile matter. He did

not believe it possible to determine water accurately in such substances by any of the methods which had been proposed and adopted in the association. The tendency of levulose to decompose even at 100° seems to render necessary some method of determination at a lower temperature. Perhaps the best that could be done would be to employ a comparative method which should be followed by all the chemists, so that so far as comparative work was concerned the results would be sufficiently near together.

Mr. Ross had recently seen a report of some investigations by Herzfeld, of the laboratory of the German Beet Sugar Association, which tended to show that at the end of fifty-six hours' drying in vacuum of a chemically pure sirup there was still a loss in weight, while by the ordinary method of drying there was secured at the expiration of the same time 1½ per cent more water than was actually present in the solution originally prepared.

The recommendations were again read, and were severally adopted.

On motion of Mr. Parsons, and after some discussion, it was agreed that the reporter on nitrogen be allowed to change the method of standardization of acids so that the chemist might either measure or weigh his acid solution for standardization, and that the words "alternate method" should no longer apply to the Gunning method.

Mr. WILEY. Mr. Roessler, one of the Austrian chemists, has sent to me, for presentation to the association in connection with the report on wines, a paper on the official methods proposed to be used in the examination of wines in Austria. I would ask that, as a matter of courtesy, I be given leave to print this valuable paper in the bulletin. I make a motion to that effect.

The motion was agreed to.

METHODS PROPOSED FOR ADOPTION FOR USE IN AUSTRIA FOR THE ANALYSIS OF WINES, BEERS, ETC.

WINES.

A.—Grape Wines.

These are divided into so-called natural wines, full wines, and half wines.

(1).—NATURAL WINES AND FULL WINES.

(1) *Definition.*—Natural wines or wines are the product obtained by the alcoholic fermentation of the juice of the grape.

(2) *Constituents.*—The most important constituents of natural wines are: Water; ethyl alcohol, compound ethers, free acids, namely: (*a*) volatile (acetic and carbonic acids); and (*b*) nonvolatile (tartaric, malic, tannic, and succinic) acids; salts of these acids; grape and fruit sugar; glycerol; nitrogenous constituents; coloring matter; mineral matter, especially potassium, sodium, calcium, magnesium, iron, aluminum, phosphoric acid, sulphuric acid, chlorine, silicic acid.

(3) *Allowable additions.*—The allowable additions before or after fermentation are: (*a*) Fresh or dried grapes, fresh or concentrated grape-juice; (*b*) Spirit free from fusel oil or cognac, and this in the following proportions: In general, the addition of alco-

hol must not amount to more than 2 volume per cent, nor the total alcohol content of the wine to more than 15 volume per cent. Exceptions are made in the case of those wines whose mode of preparation results in a higher percentage of alcohol. This in the case of sweet Tokay amounts to 16 volume per cent, in fermented Tokay to 18 volume per cent, Malaga 18 per cent, Greek, Cyprian, Asiatic, California, and Cape wines to 20 volume per cent, Madeira 21 per cent, Australian wines 21 per cent, Port wines 23 per cent, Sherry and Marsala 25 volume per cent. In Sicilian wines, a maximum of 27 volume per cent was only found exceptionally; (c) Pure cane or invert sugar; (d) pure carbonic acid; (e) pure calcium carbonate; (f) clarifying agents not injurious to health, such as tannin, albumen, gelatin, hausenblase, kaolin, talcum. If, however, by the addition of any of the materials named in (a)–(f) such changes are caused in the wine that it becomes of the character of a half wine it must be designated as such; (g) other substances added for certain purposes are only permissible under the following conditions: (1) That these by their nature and amount are not injurious to health; (2) that no material increase in the amount of wine is caused by them; (3) that each individual addition is clearly designated.

These additions include those that are made for dietetic or preserving purposes, such as pure honey, malt, saccharin, bitterwort, fruit juice, and fruit wines.

Wines with medicinal additions are medicines and are not included in the law against the adulteration of foods. Only such wines can be used, however, for their preparation as well as for curative purposes as comply fully with the regulations for pure grape wines, contain no SO_2 and not more than one grain of potassium sulphate per liter. These requirements also apply to all those wines which are sold as medicinal wines as well as to those which are used for dietetic purposes. In all cases where these requirements are not complied with the wines must be designated as injurious to health or with half wines as adulterated.

(4) *Non-permissible additions.*—The non-permissible additions before or after fermentation are aluminum and magnesium preparations, gypsum, sulphites, mineral acids, coal tar colors as well as foreign colors in general, starch, sugar, molasses, impure cane and invert sugar, impure alcohol, salicylic acid, glycerol, and artificial flavorings as well as injurious substances in general.

(5) *Chemical examination.*—For the examination and judgment the following rules adopted by the international agricultural and forestry congress at Vienna, 1890 (see Ztschr. für Nahrungsmittel-Untersuchung, Hygiene und Waarenkunde, December, 1890), are to be followed in general; for the Austro-Hungarian sweet wines, which are recommended as sanitary wines, the resolution adopted by the congress of food chemists and microscopists, Vienna, 1891 (see Ztschr. für Nahrungsmittel-Untersuchung, Hygiene und Waarenkunde, December, 1891, p. 840).

The chemical examination must always be preceded by a general examination, and the result with regard to the condition, color, odor, taste, and eventual characterization of the variety of wine must be shown in the certificate. The wine to be examined must be filtered if necessary, freed of carbonic acid, and all measurements must be made at 15°C. Each wine must be examined microscopically with a view to observe decomposition; but especially the following phenomena are to be observed: (1) Kahm or kuhnen of the wine, (2) presence of acetic ferment, (3) lactic acid fermentation, (4) glycerol fermentation, (5) coagulation, (6) stringy condition of the wine, (7) formation of astringent matter, (8) formation of brown or black colorations.

Wines exhibiting any one or more of these phenomena are to be designated as decomposed or imperfect, and the form of decomposition as well as the result of the microscopical investigation must be shown in the certificate.

(1) The specific gravity must be determined at 15° C. with the pycnometer. In general the specific gravity must be given accurately within four decimal places.

(2) The analytical results must be represented as grams per 100 cc. (eventually per one liter), and if possible in per cents by weight; only the alcohol content is to be given in value per cent.

(3) The determination of the alcohol is made by the distillation method, the sp. gr. of the weighed distillate at 15° C. with the pycnometer and the alcohol content calculated therefrom.

(4) Extract.—(a) In wines which contain not more than 3 grams extract per 100 cc., 50 cc., are evaporated in a flat platinum dish on the water bath, the residue dried in a water oven for two and a half hours, cooled in a desiccator, and weighed; (b) Wines containing a higher percentage of extract must be diluted with water to such an extent that they do not contain more than 3 per cent per 100 cc. The residue from 50 cc. of the diluted wine is determined as in (a); (c) For sweet wines the indirect method of determining the extract from the specific gravity of the dealcoholized wine is to be preferred. Balling's table must be used for calculating the extract from the specific gravity.

(5) The amount of free acid present is to be determined by titration with a suitable diluted and standardized solution of sodium or potassium hydrate, using litmus paper as an indicator. It must be stated as tartaric acid ($C_4H_6O_5$). Volatile acids are determined by distilling in a current of steam, and are expressed as acetic acid ($C_2H_4O_2$).

(6) The determination of the glycerol is to be made until further action by the Börgmann method.

(7) The mineral matter is determined by igniting the residue from 50 cc. of wine.

(8) The polarization must be made especially with sweet wine, and if necessary before and after inversion and after fermentation with washed beer yeast. Even with sour wines the polarization is a good indication of the sugar content of not completely fermented wines.

(9) The sugar is determined according to Soxhlet and is expressed as invert sugar. In the presence of much sugar, or when the difference between the extract and the directly reducing sugar is considerable, another determination must be made after inversion.

(10) Sulphuric acid is determined directly in the wines by means of barium chlorid after slightly acidifying with hydrochloric acid.

(11) The determination of sulphurous acid is made by distilling the wines to which phosphoric acid has been added in a current of carbonic acid gas, collecting the distillate in iodin solution and determining the sulphuric acid formed. Ripper's method may also be used. (See J. prakt. Chem., vol. iv, p. 425.)

(12) Phosphoric acid is determined by the molybdate method.

(13) Potassium bitartrate (argol) is determined by the method proposed by Nessler and Barth (precipitation with alcohol).

(14) Free tartaric acid is determined only when it is necessary for the completion of the other results. A simply qualitative proof of its presence is of no value for the judgment of the wine. For the quantitative determination of the tartaric acid simultaneously with the potassium bitartrate, the method proposed by B. Haas (Ztsch. für Nahrungsmitteluntersuchung und Hygiene, 1888, p. 100) may be used.

(15) Examination for coloring matters: (a) Vegetable colors. An excess of lead acetate is added to the wine and the precipitate filtered off. With genuine red wine the precipitate can be grayish-blue, bluish-gray, gray, or greenish. If with the wine under examination we obtain a precipitate which is not considerably different in color from these shades, it is unnecessary to test further for foreign vegetable colors, as the result would be very uncertain. The coloring matter of the huckleberry gives a blue precipitate with lead acetate; that of the elderberry a green precipitate. Only the coloring matter of the pokeberry (phytolacca) is radically different in its behavior from that of red wine in giving a reddish-violet precipitate. (b) Coal-tar colors: As the coloring of wines with coal-tar colors is strictly forbidden, it is sufficient to prove their presence. In most cases the presence of a coal-tar color can be detected in a wine by the following methods: (1) By shaking the filtrate from the lead precipitate with amyl alcohol and testing the latter; it is, however,

necessary to precipitate 100 cc. of wine with 30 cc. of lead acetate solution. (2) By the wool test, according to Strohmer (Archiv. für Hygiene, 1883), or Arata (Repertorium für analytische Chemic., 1887, p. 264). (3) By the mercuric oxid test, according to Cazeneuve (Vjschr. Chem. Nahr., 1886, p. 80). (4) By the method proposed by the German commission of shaking the wine with ether before and after saturation with ammonia. (5) By a similar treatment with amyl alcohol.

(16) Salicylic acid is best detected by shaking the wine with carbon bisulphid or with a mixture of ether and petroleum ether and testing the residue left upon evaporation of the solvent with ferric chlorid. When much more than 100 cc. of wine are used, a slight coloration can be caused by the normal constituents of the wine.

(17) Saccharin is detected by the method recommended by Schmitt. The wine is shaken with a mixture of ether and petroleum ether, the residue left upon evaporating the solvent fused with sodium hydrate and tested for salicylic acid. Not more than 100 cc. of wine must be used for this test.

(18) Nitric acid is found by means of a solution of diphenylamin in concentrated sulphuric acid. From one to three drops of wine are added, the usual precaution with regard to impurities being observed. For the details of the above methods I refer to the commentary.

(2).—HALF WINES.

(1) *Definition.*—Half wines are a product containing wine which is obtained by increasing the grape juice, or a natural wine by the addition of water, or water and other substances, or in a similar manner from the pomace or Weingeläger. This is also true for the products obtained from dried grapes or concentrated grape juice by the addition of water. The products obtained by diluting the grape juice with water and adding alcohol or sugar, by mixing the pomace with sugar solution and fermenting, or by extracting the pomace with dilute alcohol, are included in this list.

(2) *Constituents.*—They are in general the same as those given under I (2) for natural wines, though usually present in a different proportion.

(3) *Permissible additions.*—The permissible additions before and after fermentation are in general the same as those given for natural and full wines. Half wines can, in no case, however, be used for medicinal or dietetic purposes.

(4) *Forbidden additions.*—The same as those stated in the case of natural wines.

(5) *Examination and judgment.*—For their examination and judgment the principles laid down by the International Agricultural and Forestry Congress in Vienna, 1890 (see Zeitschrift für Nahrungsmitteluntersuchung, Hygiene und Waarenkunde, December, 1890), are to be followed.

B.—BERRY AND OTHER FRUIT WINES.

(1) *Definition.*—Berry, or fruit wines in general, are wines obtained by the alcoholic fermentation of fruits or their juice with or without the addition of alcohol, sugar, or water.

(2) *Constituents.*—Together with the majority of those given under grape wines, other constituents which are given in the chapter on ciders and fruit juices, and which are peculiar to the fruits used, occur.

(3) *Permissible additions.*—The permissible additions before or after fermentation are the same as with grape wines, with the exception of calcium carbonate.

(4) *Forbidden additions.*—The forbidden additions before or after fermentation are the same as with grape wines.

(5) *Examination.*—For their examination the analytical methods prescribed for full wines are to be used.

The principles laid down for the judgment of grape wines can only be used conditionally, as we have to consider the individual characteristics of the fruits used.

C.—Artificial Wines.

(1) *Definition.*—Artificial wines are wine-like products obtained from a mixture of different substances, imitating the wine or the grape juice.

(2) *Constituents;*

(3) *Permissible additions;*

(4) *Forbidden additions;* and

(5) *Methods of examination and judgment.*—These are in general the same as with full and half wines though again with the restriction that artificial wines must never be used for medicinal or dietetic purposes.

(6) *Designation.*—Each artificial wine which is sold in commerce must be designated as such.

D.—Miscellaneous.

All other products found in the trade which are called wine, "such as rice wine, palm wine, malt wine, birch wine, pulque," etc., must be clearly and explicitly labeled or designated as such.

GRAPE JUICES.

(1) *Definition.*—The juice obtained from the mashed grapes without any addition. Grape juice which is partially fermented may still be designated as such as long as less than one-half of the sugar present has fermented.

(2) *Constituents.*—The most important constituents of grape juice are: Water, grape or fruit sugar, free acids (mostly tartaric and malic acid and only very little tannic acid), salts of these acids, nitrogenous constituents, gums, pectinous substances, coloring matter, mineral matter (mostly potassium, sodium, calcium, magnesium, iron, aluminum, phosphoric acid, sulphuric acid, chlorin, and silicic acid).

(3) *Permissible additions.*—The permissible additions are dried grapes, concentrated grape juice, pure cane or invert sugar (eventually pure alcohol not more than 2 per cent by volume), pure calcium carbonate. Other additions (malt, honey, saccharin and similar sweetening substances, bitterwort, fruit juices, mustard seed, spices and similar substances) must be clearly designated on the label. If this is omitted a juice to which such substances have been added must be considered adulterated.

(4) *Forbidden additions.*—The forbidden additions are aluminum and magnesium preparations, gypsum, sulphurous acid, sulphites, mineral acids, coal-tar colors, starch sugar, molasses, impure cane and invert-sugar, impure alcohol, glycerol, salicylic acid, as well as injurious substances in general.

(5) *Examination of grape juice.*—The juice to be examined must be filtered. (*a*) The specific gravity is taken with a pycnometer at the normal temperature 17.5° C. The results are stated in grams per 100 cc., eventually in one liter, and, if possible, as per cents by weight. (*b*) The extract is determined by taking the amount of cane sugar corresponding to the specific gravity in Balling's table. It may also be obtained by means of a carefully tested saccharometer, which is divided into tenths. (*c*) The determinations of the free acid, potassium bitartrate, free tartaric acid, sugar, nitrogen, mineral matter, phosphoric acid, sulphuric acid, and sulphurous acid, as well as the examinations for saccharin and salicylic acid, starch sugar, and cane sugar, are made by the methods prescribed in the examination of wines.

(6) *Genuineness.*—The following points can serve to determine the genuineness of a grape juice: (*a*) The minimum amount of non-sugar obtained by subtracting the sugars calculated as invert sugar from the total extract is 16 grams per liter. (*b*) The minimum extract residue obtained by deducting from the total extract the sugars calculated as invert sugar and the free acid calculated as tartaric acid is 8 grams per liter. (*c*) The polarization corresponds either to the sugar content calculated as invert sugar or the rotation is to the left. (*d*) The amount of potassium bitartrate present is usually not less than 4 grams per liter. A partially fermented

juice contains approximately as much potassium bitartrate as a mixture of water and alcohol, which contains as much of the latter as the fermenting juice is able to dissolve. (e) The minimum amount of ash is 2 grams per liter.

With regard to the use of the analytical data we must remember that standards are only used in lieu of something more satisfactory and must never be applied comparatively; especially is this true of the values derived from the results of chemists in one country, which can only be used with the utmost caution in judging wines from other countries.

General principles.—(a) For the judgment of individual wines from certain locations, years, and varieties of grapes, only analyses of the same wines from the same locations, years, and varieties of grapes can be used as a standard. (b) The general points given in the following paragraphs denote without considering the location, variety of grapes, and year, only the extreme limits within which we may consider a wine as a natural or full wine.

(1) *Extract.*—In completely fermented wines which no longer contain an estimable amount of sugar the minimum amount of extract according to previous experience is 14 grams per liter. Wines whose per cent of extract after deducting the sugar is less than 14 grams per liter are therefore to be suspected as long as it is not proved that other natural wines of the same location and year occur with a similarly small amount of extract.

(2) *Extract residue.*—After deducting the non-volatile acids the extract residue in natural wines as a rule is not less than 11 grams per liter. Wines which contain a smaller extract residue are, however, not to be suspected as long as the results of the other investigations do not justify it.

(3) *Mineral matter.*—Wines which contain less than 1.3 gram mineral matter per liter are to be suspected as long as it is not proved that natural wines of the same location and year and similarly prepared contain an equally small amount of mineral matter.

(4) *Ratio of mineral matter to extract.*—Wine in which the mineral matter amounts to more than 10 per cent of the extract generally contains a higher percentage of extract than has been adopted as a minimum. In natural wines the mineral matter and extract frequently approach the ratio of 1 to 10 by weight. A considerable deviation from the proportion does not, however, justify the assumption that the wine is adulterated.

(5) *Ratio of alcohol to glycerol.*—The ratio between alcohol and glycerol in sour wines usually varies from 100 parts by weight of alcohol to 7 parts by weight of glycerol and 100 parts by weight of alcohol to 14 parts by weight of glycerol. If a wine shows a different ratio we can conclude that either alcohol or glycerol has been added. As during the treatment in the cellar small quantities of alcohol may get into the wine, we have to take this into consideration when judging it. Wine in which the addition of glycerol has been proved must be unconditionally rejected.

(6) *Free tartaric acid.*—In natural wines containing as much as 8 grams free acid in a liter, the amount of free tartaric acid, according to past results, is not more than one-sixth, with a maximum of one-fifth of the non-volatile acids. In natural wines containing more free acid, the amount of free tartaric acid is often much higher. Wines with an acid content of 8 grams per liter which contain more than the given amount of free tartaric acid are to be rejected either when the quantity of the latter is exceptionally high or when the other results obtained justify it (for instance if nitric acid is present).

(7) *Sodium chlorid.*—Wines which contain more than 0.05 per cent sodium chlorid are, as a rule, to be rejected. Exceptions may occur in wines which grew on ground rich in sodium chlorid.

(8) *Sulphuric acid.*—Wines which contain more than 0.92 grams sulphuric acid (SO_3), corresponding to 2 grams potassium sulphate per liter, are to be designated as having had their percentage of sulphuric acid increased by the addition of gypsum or in some other manner.

(9) *Phosphoric acid.*—The assumption that the better varieties of wines contain more phosphoric acid than the poorer is without foundation. Nevertheless the percentage of phosphoric acid present may in many cases be of great value in judging wines, especially with medicinal wines and those from certain locations in which the amount of phosphoric acid generally varies within well-defined limits. Austrian and Hungarian sweet wines, which are recommended as sanitary wines, should contain at least nearly 0.05 grams phosphoric acid (P_2O_5), in which case the other constituents must, however, be present in correspondingly proper amounts.

(10) *Nitrogen.*—The nitrogen content in sweet wines, according to the results obtained at the royal experiment station at Klosterneuburg, amounts rarely to less than 0.07 grams per liter, is usually not more than 0.8 grams per liter, and can in solitary cases be higher and rise to 1.35 grams per liter. Natural wines which do not contain more than 0.8 grams per liter have, as a rule, not only a correspondingly high sugar-free extract, but prove themselves by their taste to be of good quality. Wines which lack these properties are either spoilt or Hefepresz-Geläger oder Hefeweine. Wines which contain less than 0.07 grams nitrogen per liter are, however, only to be rejected when this is justified by the other results.

(11) *Nitric acid.*—Wines in which the presence of nitric acid has been proved are only to be rejected when the amount of nitric acid present (according to the intensity of the diphenylamin reaction) is exceptionally high or when, in connection with a small amount of nitric acid, some other analytical results speak for a dilution of the wine with water.

(12) *Sulphurous acid.*—When, in finally judging a wine, we are not limited to deciding whether it is a natural wine or not, but must also determine the presence of injurious substances, the amount of sulphurous acid present must be given.

FRUIT JUICES, FRUIT SIRUPS, FRUIT JELLIES, ETC.

(1) *Definition.*—By fruit juices we mean the liquid obtained from mashed fruits without any addition whatever. The fruit juices of the trade and of the home are liquids which are obtained by the action of preserving agents upon pure fruit juices.

The methods of preservation used in their preparation depend on alcoholic fermentation, application of heat, boiling, concentration, sterilization, evaporation, use of preserving agents, sugar, alcohol, spices.

Fruit juices which show a high percentage of sugar (50 per cent and over) and are correspondingly thick are usually called fruit sirups. Fruit jellies are fruit juices of the trade which have been concentrated by heating and subsequently gelatinized on cooling.

(2) *Constituents of pure fruit juices.*—In the pure fruit juices obtained from ripe fruits the following substances have been detected so far: Water; free acids, namely, malic acid (in the juice of most fruits); citric acid (in the juice of the lemon, orange, cherry, currant, gooseberry, raspberry, etc.); tannic and tartaric acids: benzoic acids (in the juice of the pineapple, tamarind, etc); salts of these acids; grape and fruit sugar; cane sugar (in the juice of many apples and some pears, as well as in apricots, plums, peaches, huckleberry, strawberry, lemons, oranges and pineapple); gums; pectinous bodies; nitrogenous constituents; coloring matter; aromatic bodies; ash (mostly potassium, sodium, calcium, magnesium, iron, manganese, phosphoric acid, sulphuric acid, chlorin and silicic acid.)

(3) *Permissible additions.*—Substances which may be added in the preparation of fruit juices for the trade are: Clarifying agents not injurious to health, such as gelatin, albumen, kaolin, talcum and tannin, alcohol free from fusel oil, cognac, brandy, pure cane or invert sugar, spices, pure sodium chlorid, pure acetic acid; and—

when their addition is clearly designated—malt extract, honey, saccharin, and similar sweetening substances.

(4) *Forbidden additions.*—The forbidden additions are: Coal tar colors and other injurious coloring matters, starch sugar, molasses, impure cane or invert sugar, impure alcohol, aluminum or magnesium preparations, gypsum, sulphurous acid and sulphites, mineral acids, salicylic acid, glycerol, artificial flavors, ethers, injurious substances in general.

(5) *Chemical examination.*—(*a*) The fruit juice to be examined must, if necessary, be diluted and filtered. (*b*) Specific gravity determined at 17.5° C. (*c*) The analytical results must be stated in per cents by weight, eventually as grams per 100 cc. (or in one litre), the alcohol as volume per cent. (*d*) All samples for analysis are weighed, not measured. (*e*) Alcohol, extract, free acid, volatile acids, glycerol, nitrogen, sugar, ash, sulphuric acid, sulphurous acid, phosphoric acid, as well as the polarization and the presence of coloring matters, salicylic acid, and saccharin are determined by the methods given in the chapter on "Wine." (*f*) The examination for heavy metals (copper, etc.) can be made in the ash, of the fruit juice. If a general examination for heavy metals is, however, required, the organic constituents must be destroyed in the wet way.

(6) *Judgment of fruit juices.*—For this the following points have the greatest weight: (*a*) The result of the preliminary examination by means of which condition (degree of transparency) color, odor, and taste are determined. Opaque, discolored, foul smelling, and bad tasting fruit juices are naturally not fit for consumption and must be rejected. (*b*) The results of the chemical examination in connection with the determinations in sections 3 and 4. Outside of the substances named in section 4 fruit juices should contain no poisonous heavy metals (lead, copper, zinc). The results of previous analyses of pure fruit juices or the fruit juices of the trade.

Mr. WOLL made a motion, which, as amended in discussion and adopted, is as follows:

Moved, That a committee of five be appointed by the chair during the first session of each convention of the association, to whom all recommendations of the various reporters and all changes in methods proposed by members of the association shall be referred, and who, after consultation with the reporters and members proposing changes concerning the desirability of adopting such recommendations and changes, shall report those concurred in to the association: *Provided,* That any member shall have the privilege of calling up before the association such recommendations and changes as are not concurred in by the committee; and these may then be passed by a two-thirds majority vote of the members present.

On motion of Mr. WILEY, the following paper was ordered to be printed:

SUGGESTIONS FOR THE BETTER ARRANGEMENT OF FOOD EXAMINATIONS.

By Dr. WILLIAM DICKORÉ.

We all know that in the legislatures of our States, as a rule, very little scientific talent is to be found, and consequently the law by which the work of the food commissioners is governed is frequently ridiculous, if not hurtful, to the legitimate interests of individuals as well as to that of the broad masses. There are especially three articles to which this refers, namely, oleomargarine, vinegar, and certain classes of jellies.

Before I go any further let me state that what I have to say will not be said with the desire to protect fraudulent dealers who sell these goods for anything else than what they are, but in the interest of these articles as wholesome and useful substitutes for direct natural products, to which they in purity and cleanliness are sometimes superior; and it will be said in defiance of prejudice and ignorance.

OLEOMARGARINE.

It is not necessary to repeat to a body of scientific and practical chemists the processes by which this article is produced. I will only call to the minds of these gentlemen that it is the life interest of every manufacturer to obtain raw material that is fresh and wholesome, to keep it during the process of manufacture in that state, and to prepare his product so that it will stay in that condition a long time—this latter because he produces large quantities and he would be the loser if it should spoil in an unreasonably short time. On the other hand, dairy butter is manufactured mostly on a small scale and neither producer nor consumer expects it to keep very long; therefore the producer runs less pecuniary risk if it is impure and carries the germ of decomposition (casein and whey) in it.

We all know that most of the small butter producers are very unscrupulous in that respect, and in buying butter we frequently pay the butter prices for over 20 per cent of casein, whey, and salt, and have an article which will spoil in a few days, turning rancid and unwholesome; and not all consumers can afford to throw butter away when it gets in that state. Pure oleomargarine, on the other hand, being free of azotized substances, will not undergo such rapid decomposition but remains wholesome for a long time.

Under such circumstances I can not see the benefit which the poorer classes derive from the State laws, which, by prohibiting the addition of harmless coloring matter to it, makes the honest sale of oleomargarine almost impossible. The consumer of a butter substitute does not look for the taste alone; he also wants that yellowish color to gratify his eye as well as his palate. Would anybody enjoy a cup of the best Mocha coffee if the brown color had all been removed from it?

VINEGARS.

The same question of color refers to certain vinegars. We all know that vinegar obtained by fermentation of grain, distillation of the alcohol thus produced, and oxidation of this alcohol by the generator process is perfectly healthful; in fact, much cleaner and more free from possible impurity and danger to health than wine or cider vinegars. But it is colorless, and the public, accustomed for centuries to see the vinegars produced from fruit juices of a more or less brown color, has a prejudice against colorless vinegars, calls them artificial, and believes them to be made out of wood or mineral acids, and to be impure and unwholesome. They do not know that the brown color of cider vinegar is due to impurities only, coming from iron and the tannic principles or from contact with the wood of the barrel stave. This being true, why should the vinegar manufacturer be prohibited from giving his colorless product the desirable shade by adding a few crumbs of burnt sugar?

JELLIES.

Another article, which at reasonable prices fills a real demand, is a certain class of jellies produced by congealing grape sugar with tartaric acid. These ingredients are absolutely harmless to health, the product is pleasant to eat, and the poorer classes, who can not afford to indulge in jellies for from thirty to fifty cents or more per pint, get a substitute which gratifies their palate without hurting either their stomachs or their pocketbooks.

The consumers are contented and would remain so if it were not for Mr. Health Officer or Mr. Food Commissioner, who is burning with a desire to see his name glorified in the daily papers as a benefactor of the human race. Learning from his good housewife that it is an absolute impossibility to sell for such low prices jelly prepared from fruit juice and cane sugar exclusively, he starts out to collect samples and has them examined by the chemist of his department. These gentlemen, of course, get at the bottom of this thing and report the greater or smaller absence of real fruit juice, but very seldom or never do they pronounce, in their report, that the sample,

while it is, perhaps, not what it is called on the label, is still an absolutely harmless and delightful article of diet. A few days afterwards we see in the newspapers long articles, under big headlines, about the "impure food," "adulterated food," etc. A cold shudder runs down the spinal cord of the public in general, and everybody is surprised that he is not dead and gone long ago when such imminent dangers seem to lurk in the food which he has enjoyed for many years with good appetite and health.

If the rattled reader of the "impure food" articles would also look over the health and mortality statistics of civilized nations he would be surprised to see that the age which produces such artificial substitutes is not lacking in general health and longevity, but that a marked increase for the better is noticeable.

But all these facts are well known to the members of the chemical society, and I would like to mention the purpose for which I have called these examples to your attention. In some States the food laws are made exclusively for the protection of the farmer, at the expense of industry and the progress of human ingenuity; in other States, the public may be injured to favor special interests of other classes, and I think it should be one of the objects of this association to make an effort in the direction of protecting the public, not only against the fraud which is exercised by unscrupulous manufacturers and dealers who produce unhealthful articles, or those who pass artificial ones for genuine, but also against the unscrupulous or ignorant persons who unjustly terrify and mislead the public about such articles of food as mentioned above, with the intention of depriving them of the position they deserve in our national economy.

A CENTRAL FOOD COMMISSION.

Therefore, let us use our influence, the influence of unprejudiced scientific men, that the Federal Government, alone, shall take the food question into its strong hand; that it may create a Central Food Commission at Washington, with branch stations for certain districts, independent of local political considerations; that it be the aim of this commission, not only to hunt up and drive out of the market unwholesome goods, but also to protect others which are persecuted and suppressed wrongfully by local interests, prejudice, or ignorance; that it also be the object of this commission to educate the people by giving, along with the analyses of the chemists, statements whether the respective articles of food are injurious to health or not; that, to facilitate this, it shall have the right to compel manufacturers to lay open to the commission their processes of manufacture and quality and kind of raw materials used; that it be compelled to give certificates as to healthfulness of goods, by which certificates the manufacturer may sell in all States of the Union without hindrance by State authorities; that this commission be compelled to be careful not to arouse the sentiment of the masses against the progress of science in its aim to improve products of nature, or to produce harmless substitutes artificially, where the common natural sources are not sufficient to fill the demand; and that the farmers' products shall be subject to the same strict rules and regulations as any manufactured goods.

I imagine that, if such a commission should be created on this or a similar basis, it would be a great benefit to all classes of our population.

NOTICE OF THE DEATH OF DR. N. T. LUPTON, EX-PRESIDENT OF THE ASSOCIATION.

Mr. WILEY. Since our last meeting one of our oldest and most distinguished members, an ex-president of this association, has died, and it seems to me appropriate that we should take action expressing our appreciation of the services he has rendered. Dr. Lupton was present

in Atlanta in May, 1884, when a meeting was held out of which grew the organization of this association at Philadelphia in September of the same year. In a sense he was a charter member of the association. He attended all our meetings, and at each of them he contributed valuable work to the proceedings. As I have said, he was our president. I shall move that, in honor of his memory, the members present rise to their feet after Mr. Ross, whom I will ask to say a few words in its support, has spoken.

Mr. Ross. I most heartily second the motion made by Mr. Wiley that this association shall manifest its sorrow for the death of our ex-president, Dr. Lupton, who was for so long a time one of our most valued members, and a man who took a very prominent part in the work of several of the leading scientific associations of this country, and filled, with marked ability, a number of very important positions in the line of chemistry.

The motion was unanimously carried, and the members present rose in honor of Dr. Lupton.

Mr. Frear. I would like to have an interpretation by the president as to the effect of a motion I made last year in regard to the appointment of a committee to which should be relegated the work of abstracting current literature. Was that understood to be continuous in effect, or was it only for last year?

The President. I understood it was to be continuous; that an abstract committee was to be appointed each year.

Mr. Frear. I move that the association tender to its honorable secretary its warm sense of indebtedness for his constant care, which has so largely contributed to the ends we have been endeavoring to attain in our work as an association.

The motion was agreed to.

On motion, at 1:30 p. m., the convention adjourned.

Note.--During the unavoidable absence of the editor in Chicago, Mr. G. L. Spencer read the proof of this bulletin, assisted by Mr. K. P. McElroy. The editor desires to thank these gentlemen for their painstaking labors.—H. W. W.

OFFICERS, REPORTERS, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS OF THE UNITED STATES FOR 1893-'94.

PRESIDENT.

Mr. E. B. VORHEES, chemist of the New Jersey Experiment Station.

VICE-PRESIDENT.

Mr. H. A. HUSTON, State chemist of Indiana.

SECRETARY.

Mr. H. W. WILEY, Chief Chemist U. S. Department of Agriculture.

EXECUTIVE COMMITTEE.

The president, vice-president, and secretary, *ex officio.*
Messrs. B. B. Ross, of Auburn, Ala.
C. D. WOODS, of Middletown, Conn.

REPORTERS.

Phosphoric acid.—B. W. Kilgore, Raleigh, N. C.
Nitrogen.—J. M. Bartlett, Orono, Me.
Potash.—H. J. Wheeler, Kingston, R. I.
Soils and ash.—A. M. Peter, Lexington, Ky.
Dairy products.—E. H. Farrington, Champaign, Ill.
Foods and feeding stuffs.—H. J. Patterson, College Park, Md.
Fermented liquors.—C. A. Crampton, Washington, D. C.
Sugar.—G. L. Spencer, Washington, D. C.

ABSTRACT COMMITTEE.

William Frear, State College, Pa.
E. W. Hilgard, Berkeley, Cal.
W. C. Stubbs, Audubon Park, New Orleans, La.
W. D. Bigelow, Washington, D. C.
John P. Street, New Brunswick, N. J.

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CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects shall be (1) to secure uniformity and accuracy of the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary who shall also officiate as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a reporter for each of the subjects to be considered by the association.

It shall be the duty of these reporters to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(8) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(9) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present entitled to vote.

OFFICIAL METHODS OF ANALYSIS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT ITS MEETING, AUGUST 24, 25, 26, 1893.

METHODS FOR PHOSPHORIC ACID.

PREPARATION OF SAMPLE.

The sample should be well intermixed, finely ground, and passed through a sieve having circular perforations 1 mm. in diameter. The process of grinding and sifting should take place as rapidly as possible, so that there should be no loss or gain of moisture during the operation.

DETERMINATION OF MOISTURE.

(*a*) In potash salts, sodium nitrate, and ammonium sulphate, heat from 1 to 5 grams at 130° until the weight is constant, and determine the loss in weight as moisture. (*b*) In all other fertilizers, heat 2 grams, or 5 grams if the sample be very coarse, for five hours, at 100°, in a steam bath.

METHODS OF DETERMINING PHOSPHORIC ACID.

Water-soluble phosphoric acid.—Place 2 grams in a 9 cm. filter; wash with successive small portions of water, allowing each portion to pass through before adding more, until the filtrate measures about 250 cc. If the filtrate be turbid, add a little nitric acid. Make up to any convenient definite volume; mix well; take any convenient portion and determine the phosphoric acid as under total phosphoric acid.

Citrate-insoluble phosphoric acid.—Heat 100 cc. of strictly neutral ammonium-citrate solution of 1.0900 specific gravity to 65° C. in a flask placed in a bath of warm water keeping the flask loosely stoppered to prevent evaporation. When the citrate solution in the flask has reached 65° C., drop into it the filter containing the washed residue from the water-soluble phosphoric-acid determination, stopper tightly with a smooth rubber stopper, and shake violently until the filter paper is reduced to a pulp. Place the flask back into the bath and maintain the water in the bath at such a temperature that the contents of the flask will stand at exactly 65° C. Shake the flask every five minutes. At the expiration of exactly thirty minutes from the time the filter and residue were introduced, remove the flask from the bath and immediately filter as rapidly as possible. Wash thoroughly with water at 65° C. Transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed, add from 10 to 15 cc. of strong hydrochloric acid, and digest until all phosphate is dissolved. Dilute the solution to 200 cc. If desired, the filter and its contents can be treated according to methods 1, 2, or 3 under total phosphoric acid. Mix well; filter through a dry filter; take a definite portion of the filtrate and determine phosphoric acid as under total phosphoric acid.

In case a determination of citrate-insoluble phosphoric acid is required in non-acidulated goods it is to be made by treating 2 grams of the phosphatic material,

without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.) the residue insoluble in ammonium citrate is to be treated by one of the processes described below under "total phosphoric acid" 1, 2, or 3.

Total phosphoric acid.—In case of ignition the ignited material is to be dissolved in hydrochloric acid. Weigh 2 grams and treat by one of the following methods: (1) Evaporate with 5 cc. of magnesium nitrate, ignite, and dissolve in hydrochloric acid. (2) Boil with 20 to 30 cc. of strong sulphuric acid, adding 2 to 4 grams of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. A Kjeldahl flask graduated to 250 cc. is recommended. After the solution is colorless, add 150 cc. of water and boil for a few minutes, cool, and make up to volume. (3) Digest with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning methods for estimating nitrogen. Do not add any potassium permanganate, but after the solution has become colorless add about 100 cc. of water and boil for a few minutes, cool, and make up to a convenient volume; 2.5 grams of substance and a digestion flask graduated to 250 cc. are recommended. [This method will be found convenient when both the nitrogen and the total phosphoric acid are to be determined in a fertilizer. In this case, after diluting to volume and mixing, a portion, for the estimation of nitrogen, may be removed with a pipette and the remainder then filtered through a dry filter and a portion taken for the determination of the total phosphoric acid.] (4) Solution in 30 cc. of concentrated nitric acid with a small quantity of hydrochloric acid. (5) Add 30 cc. of concentrated hydrochloric acid, heat, and add cautiously, in small quantities at a time, about 0.5 grams of finely-pulverized potassium chlorate. (6) Solution in 15 to 30 cc. of strong hydrochloric acid and 3 to 10 cc. of nitric acid. This method is recommended for fertilizers containing much iron or aluminium phosphate. Boil until all phosphates are dissolved and all organic matter is destroyed; cool and dilute to 200 or 250 cc.; mix and pass through a dry filter; take an aliquot part of the filtrate corresponding to 0.25 gram, 0.50 gram, or 1 gram, neutralize with ammonia, and clear with a few drops of nitric acid. (In case hydrochloric or sulphuric acid has been used as a solvent, add about 15 grams of dry ammonium nitrate or its equivalent.)

To the hot solutions, for every decigram of P_2O_3 that is present, add 50 cc. of molybdic solution. Digest at about 65° C. for an hour, filter, and wash with water or ammonium-nitrate solution. Test the filtrate by renewed digestion and addition of more molybdic solution. Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (about 1 drop per second), stirring vigorously. After fifteen minutes add 30 cc. of ammonia solution of density 0.95. Let stand for some time; two hours is usually enough. Filter, wash with dilute ammonia, ignite at white heat for ten minutes, and weigh.

Citrate-soluble phosphoric acid.—The sum of the water soluble and citrate insoluble subtracted from the total gives the citrate soluble.

PREPARATION OF REAGENTS.

(1) *Ammonium-citrate solution.*—(a) Mix 370 grams of commercial citric acid with 1,500 cc. of water; nearly neutralize with commercial ammonia; cool; add ammonia until exactly neutral (testing with saturated alcoholic solution of corallin) and bring to volume of 2 liters. Test the specific gravity, which should be 1.0900 at 20° C., before using.

(b) *Alternate method.*—To 370 grams of commercial citric acid add commercial ammonia, sp. gr. 0.96, until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium

chlorid, 200 grams to the liter, and add 4 volumes of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly-prepared corallin solution as preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution; 50 cc. of this solution will precipitate the citric acid from 10 cc. of the citrate solution. To 10 cc. of the nearly neutral citrate solution add 50 cc. of the alcoholic calcium-chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with neutral solution of cochineal. If acid or alkaline, add ammonia or citric acid, as the case may be, mix, and test again, as before. Repeat this process until a neutral reaction is obtained. Bring to the proper specific gravity of 1.0900 at 20° C.

(2) *Molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams or 417 cc. of ammonia, sp. gr. 0.96, and pour the solution thus obtained into 1,500 grams or 1,250 cc. of nitric acid, sp. gr. 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(3) *Ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of 2 liters.

(4) *Magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grams of ammonia chlorid, 700 cc. of ammonia of sp. gr. 0.96, and water enough to make a volume of 2 liters. Instead of the solution of 22 grams of calcined magnesia, 110 grams of crystallized magnesium chlorid ($MgCl_2$, $6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume of ammonia, sp. gr. 0.96, mixed with 3 volumes of water, or usually 1 volume of concentrated ammonia with 6 volumes of water.

(6) *Nitrate of magnesia.*—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from the excess of magnesia ferric oxide, etc., and bring to volume of 2 liters.

METHODS FOR THE DETERMINATION OF NITROGEN.

THE ABSOLUTE OR CUPRIC OXID METHOD.

[Applicable to all nitrogen determinations.]

The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 66 cm. long and 12.7 mm. internal diameter.

Azotometer of at least 100 cc. capacity, accurately calibrated.

Sprengel mercury air pump.

Small paper scoop, easily made from stiff writing paper.

REAGENTS.

Coarse cupric oxid or wire form.—To be ignited and cooled before using.

Fine cupric oxid.—Prepared by pounding ordinary cupric oxid in a mortar.

Metallic copper.—Granulated copper, or fine copper gauze, reduced and cooled in a current of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Make a supersaturated solution of caustic potash in hot water. When absorption of carbonic acid ceases to be prompt, the solution must be discarded.

Filling the tube.—Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenized substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill the tube as follows: (1) About 5 cm. of coarse cupric oxid. (2) Place on the small paper scoop enough of the fine cupric oxid to fill, after having been mixed with the substance to be analyzed, about 10 cm. of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxid. (3) About 30 cm. of coarse cupric oxid. (4) About 7 cm. of metallic copper. (5) About 6 cm. of coarse cupric oxid (anterior layer). (6) A small plug of asbestos.· (7) 0.8 to 1 gram of sodium bicarbonate. (8) A large, loose plug of asbestos; place the tube in the furnace, leaving about 2.5 cm. of it projecting; connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid (see 5th above), and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbonic acid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal, so that the escaping bubbles will pass into the air and not into the tube, thus avoiding the useless saturation of the caustic-potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the gas under the 30 cm. layer of oxid, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually the mixture of substance and oxid, lighting one jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second or a little faster.

When the jets under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxid, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and a temperature have been reached.

Adjust accurately the level of the KOH solution in bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculation as usual, or read results from tables.

THE KJELDAHL METHOD.

[Not applicable in presence of nitrates.]

(1) *Acid.*—(*a*) Standard hydrochloric acid, the absolute strength of which has been determined by precipitating with silver nitrate and weighing the silver chlorid as follows:

To any convenient quantity of the acid to be standardized: Add solution of silver nitrate in slight excess, and 2 cc. pure nitric acid, specific gravity, 1.2. Heat to boiling point, and keep at this temperature for some minutes without allowing violent ebullition, and with constant stirring, until the precipitate assumes the granu-

lar form. Allow to cool somewhat, and then pass the fluid through the asbestos. Wash the precipitate by decantation, with 200 cc. of very hot water, to which has been added 8 cc. nitric acid and 2 cc. dilute solution of silver nitrate containing 1 gram of the salt in 100 cc. of water. The washing by decantation is performed by adding the hot mixture in small quantities at a time, and beating up the precipitate well with a thin glass rod after each addition. The pump is kept in action all the time, but to keep out dust during the washing the cover is only removed from the crucible when the fluid is to be added.

Put the capsule and precipitate aside, return the washings once through the asbestos so as to obtain them quite clear, remove them from the filter, and set aside to recover excess of silver. Rinse the receiver and complete the washing of the precipitate with about 200 cc. of cold water. Half of this is used to wash by decantation and the remainder to transfer the precipitate to the crucible with the aid of a trimmed feather. Finish washing in the crucible, the lumps of silver chlorid being broken down with the glass rod. Remove the second filtrate from the receiver and pass about 20 cc. of 98 per cent alcohol through the precipitate. Dry at 140° to 150°. Exposure for half an hour is found more than sufficient, at this temperature, to dry the precipitate thoroughly.

Or (b) standard sulphuric acid the absolute strength of which has been determined by precipitation with barium chlorid and weighing the resulting barium sulphate.

For ordinary work, half normal acid is recommended; i. e., acid containing 18.2285 grams of hydrochloric acid or 24.5185 grams sulphuric acid to the liter; for work in determining very small amounts of nitrogen, one-tenth normal acid is recommended. In titrating mineral acids against ammonia solutions, use cochineal as indicator.

(2) *Standard ammonia*, the strength of which, relative to the acid, has been accurately determined. One-tenth normal ammonia solution, i. e., containing 1.7051 grams of ammonia to the liter, is recommended for accurate work.

(3) *Sulphuric acid*, specific gravity 1.84, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxids of nitrogen.

(4) *Metallic mercury* or mercuric oxid, prepared in the wet way. That prepared from mercuric nitrate can not be safely used.

(5) *Potassium permanganate* finely pulverized.

(6) *Granulated zinc*, pumice stone, or 0.5 gram of zinc dust are to be added to the contents of the flasks in distillation, when found necessary, in order to prevent bumping.

(7) *Potassium sulphid.*—A solution of 40 grams of commercial potassium sulphid in 1 liter of water.

(8) *Soda.*—A saturated solution of sodium hydrate free from nitrates.

(9) *Indicator.*—Solution of cochineal prepared as follows: Tincture of cochineal is prepared by digesting and frequently agitating 3 grams of pulverized cochineal in a mixture of 50 cc. of strong alcohol with 200 cc. of distilled water, at ordinary temperatures, for a day or two. The solution is decanted or filtered through Swedish paper.

APPARATUS.

(1) ·*Kjeldahl digestion flasks* of hard, moderately thick, well-annealed glass. These flasks are about 22 cm. long, with a round, pear-shaped bottom, having a maximum diameter of 6 cm., and tapering out gradually in a long neck, which is 2 cm. in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cc.

(2) *Distillation flasks* of ordinary shape, of 550 cc. capacity, or preferably flasks of the same capacity, of pear-shaped bottom, of well-annealed glass, for both digestion and distillation, fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation.

The bulbs are about 3 cm. in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end. This is adjusted to the tube of the condenser by a rubber tube.

(1) *The digestion.*—0.7 to 0.8 gram of the substance to be analyzed, according to its proportion of nitrogen, is brought into a digestion flask with approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury and 22 cc. of sulphuric acid. The flask is placed in an inclined position, and heated below the boiling point of the acid for from five or fifteen minutes or until frothing has ceased. If the mixture froths badly, a small piece of paraffin may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot potassium permanganate is dropped in carefully and in small quantities at a time till, after shaking, the liquid remains of a green or purple color.

(2) *The distillation.*—After cooling, the contents of the flask are transferred to the distilling flask with about 200 cc. of water, with a few pieces of granulated zinc, pumice stone, or 0.5 gram of zinc dust when found necessary to keep the contents of the flask from bumping, and 25 cc. of potassium sulphide solution are added, shaking the flask to mix its contents. Next add 50 cc. of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with acid solution. Connect the flask with the condenser, mix the contents by shaking and distill until all ammonia has passed over into the standard acid. The first 150 cc. of the distillate will generally contain all the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard ammonia.

The use of mercuric oxid in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The potassium sulphide removes all the mercury from the solution, and so prevents the formation of mercur-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present, which might otherwise escape notice.

GUNNING METHOD.

In a digestion flask holding from 250 to 500 cc. place from 0.7 to 2.8 grams of the substance to be analyzed, according to its proportion of nitrogen. Then add 10 grams of powdered potassium sulphate and from 15 to 25 cc. (ordinarily about 20 cc.) of concentrated sulphuric acid. Conduct the digestion as in the Kjeldahl process, starting with a temperature below boiling point and increasing the heat gradually until frothing ceases. Digest until colorless or nearly so. (Do not add either potassium permanganate or potassium sulphid.) Dilute, neutralize, and distill as in the Kjeldahl method. In neutralizing, it is convenient to add a few drops of phenolphthalein indicator, by which one can tell when the acid is completely neutralized, remembering that the pink color, which indicates an alkaline reaction, is destroyed by a considerable excess of strong fixed alkali. The distillation and titration are conducted as in the Kjeldahl method.

KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.

[Applicable to all fertilizers containing nitrates.]

Besides the reagants and apparatus given under the Kjeldahl method there will be needed—

(1) *Zinc dust.* This should be an impalpable powder; granulated zinc or zinc filings will not answer.

(2) *Sodium thiosulphate.*

(3) *Commercial salicylic acid.*

THE DETERMINATION.

Bring from 0.7 to 1.4 grams of the substance to be analyzed into a Kjeldahl digesting flask, add to this 30 cc. of sulphuric acid containing 1 gram of salicylic acid and shake thoroughly; then add 5 grams of crystallized sodium thiosulphate, or add to the substance 30 cc. of sulphuric acid containing 2 grams of salicylic acid; then add gradually 2 grams of zinc dust, shaking the contents of the flask at the same time. Finally place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly and the boiling continued until white fumes no longer pour out of the flask. This requires about five or ten minutes. Add now approximately 0.7 gram of mercuric oxide or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless or nearly so. (In case the contents of the flask are likely to become solid before this point is reached add 10 cc. more of sulphuric acid.) Complete the oxidation with a little potassium permanganate in the usual way and proceed with the distillation as described in the Kjeldahl method. The reagents should be tested by blank experiments.

GUNNING METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.

MANIPULATION.

In a digestion flask holding from 250 to 500 cc. place from 0.7 to 2.8 grams of the substance to be analyzed according to the amount of nitrogen present. Ten grams of potassium sulphate, 5 grams of sodium thiosulphate and 30–35 cc. of the salicylic acid mixture, *i. e.* 30 cc. sulphuric acid to 1 gram of salicylic acid, are then added and the whole digested at a low temperature until frothing ceases. Then heat strongly until the solution becomes colorless or nearly so. Dilute, neutralize, and distill, as in the Gunning method.

ALTERNATE METHODS.

RUFFLE METHOD.

REAGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) A mixture of equal parts by weight of fine slaked lime and finely powdered sodium thiosulphate dried at 100°.

(3) A mixture of equal parts by weight of finely powdered granulated sugar and flowers of sulphur.

(4) Granulated soda-lime, as described under the soda-lime method.

APPARATUS.

(5) Combustion tubes of hard Bohemian glass, 30 cm. long and 1.3 cm. in diameter.

(6) Bulbed U-tubes or Will's bulbs, as described under the soda-lime method.

(1) Clean the U-tube and introduce 10 cc. of standard acid.

(2) Fit the cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of abestos, previously ignited, and then 2.5 to 3.5 cm. of the thiosulphate mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grams of the sugar and sulphur mixture. (3) Pour on a piece of glazed paper or in a porcelain mortar a sufficient quantity of thiosulphate mixture to fill about 25 cm. of tube; then add the substance to be analyzed, as previously prepared, mix carefully, and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the thiosulphate mixture and pour into the tube; then fill up with soda-lime to within 5 cm. of the end of the tube. (4) Place another plug of ignited asbestos at the end of the tube and close with a cork. (5) Hold the tube in a horizontal position and tap on the table until there is a gas channel all along the top of the tube. Make connections with the U-tube containing the acid; aspirate and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little, so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn on slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U-tube begins to recede toward the furnace attach the asspirator to the other limb of the U-tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U-tube and wash the contents into a beaker or porcelain dish; add a few drops of the cochineal solution, and titrate.

SODA-LIME METHOD.

[Not applicable in presence of nitrates.]

REAGENTS AND APPARATUS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) Granulated soda-lime, fine enough to pass a 2.5 mm. sieve and thoroughly dry.

(3) Fine soda-lime, fine enough to pass a 1.25 mm. sieve, also thoroughly dry.

Excellent soda-lime may be easily and cheaply prepared by slaking $2\frac{1}{2}$ parts of quicklime with a strong solution of 1 part of commercial caustic soda (such soda as is used in the Kjeldahl process), care being taken that there is enough water in the solution to slake the lime. The mixture is then dried and heated in an iron pot to incipient fusion, and when cold ground and sifted as above.

Instead of soda-lime Johnson's mixture of carbonate of soda and lime or slaked lime may be used.

Slaked lime may be granulated by mixing it with a little water to form a thick mass, which is dried in the water oven until hard and brittle. It is then ground and sifted as above. Slaked lime is much easier to work with than soda-lime and gives excellent results, though it is probable that more of it should be used in proportion to the substance to be analyzed than is the case with soda-lime.

(4) Asbestos which has been ignited and kept in a glass-stoppered bottle.

(5) Combustion tubes about 40 cm. long and of 12 mm. internal diameter, drawn out to a point and closed at one end.

(6) Large-bulbed U-tubes with glass stopcock, or Will's tubes with four bulbs.

MANIPULATION.

The substance to be analyzed should be powdered fine enough to pass through a sieve of 1 mm. mesh; 0.7 to 1.4 grams, according to the amount of nitrogen present, is taken for the determination. Into the closed end of the combustion tube put a

small loose plug of asbestos, and upon it about 4 cm. of fine soda-lime. In a porcelain dish or mortar mix the substance to be analyzed, thoroughly but quickly, with enough fine soda-lime to fill about 16 cm. of the tube, or about 40 times as much soda-lime as substance, and put the mixture into the combustion tube as quickly as possible by means of a wide-necked funnel, rinsing out the dish and funnel with a little more fine soda-lime, which is to be put in on top of the mixture. Fill the rest of the tube to about 5 cm. of the end with granulated soda-lime, making it as compact as possible by tapping the tube gently while held in a nearly upright position during the filling. The layer of granulated soda-lime should not be less than 12 cm. long. Lastly, put in a plug of asbestos about 2 cm. long, pressed rather tightly, and wipe out the end of the tube to free it from adhering soda-lime.

Connect the tube by means of a well-fitting rubber stopper or cork with the U-tube or Will's bulbs, containing 10 cc. of standard acid, and adjust it in the combustion furnace so that the end projects about 4 cm. from the furnace, supporting the U-tube or Will's bulb suitably. Heat the portion of the tube containing the granulated soda-lime to a moderate redness, and when this is attained extend the heat gradually through the portion containing the substance, so as to keep up a moderate and regular flow of gases through the bulbs, maintaining the heat of the first part until the whole tube is heated uniformly to the same degree. Keep up the heat until gases have ceased bubbling through the acid in the bulbs, and the mixture of substance and soda-lime has become white, or nearly so, which shows that the combustion is finished. The combustion should occupy about three-quarters of an hour or not more than one hour. Remove the heat, and when the tube has cooled below redness break off the closed tip and aspirate air slowly through the apparatus for two or three minutes to bring all the ammonia into the acid. Disconnect, wash the acid into a beaker or flask, and titrate with the standard alkali.

During the combustion the end of the tube projecting from the furnace must be kept heated sufficiently to prevent the condensation of moisture, yet not enough to char the stopper. The heat may be regulated by a shield of tin slipped over the projecting end of the combustion tube.

It is found very advantageous to attach a Bunsen valve to the exit tube, allowing the evolved gases to pass out freely, but preventing a violent "sucking back" in case of a sudden condensation of steam in the bulbs.

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METHODS OF DETERMINING POTASH.

METHOD OF LINDO-GLADDING.

(1) *In superphosphates.*—Boil 10 grams with 300 cc. of water 30 minutes. To the hot solution add ammonia in slight excess, and then a sufficient quantity of ammonium oxalate to precipitate all the lime present; cool and make up to 500 cc., mix thoroughly, and filter through a dry filter; take 50 cc., corresponding to 1 gram, evaporate nearly to dryness, add 1 cc. of dilute sulphuric acid (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. This residue is dissolved in hot water, plus a few drops of hydrochloric acid, and a slight excess of platinum solution is now added. This solution is then evaporated to a thick paste in a small dish, and 80 per cent alcohol added. In evaporating, special precaution should be taken to prevent absorption of ammonia. The precipitate is washed thoroughly with alcohol by decantation and on the filter, as usual. The washing should be continued even after the filtrate is colorless. Ten cc. of the ammonium chlorid solution, prepared as hereafter directed, are now run through the filter, or the washing may be performed in the dish. The 10 cc. will contain the bulk of the impurities, and are thrown away. Fresh portions of 10 cc. of the ammonium chlorid are now run through the filter several times (5 or 6). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. Care should be taken that the precipitate is perfectly soluble in water. The platinum solution used contains 1 gram of metallic platinum in every 10 cc. To prepare the washing solution of ammonium chlorid, place in a bottle 500 cc. of water 100 grams of ammonium chlorid; shake till dissolved. Now, pulverize 5 or 10 grams of potassium platinic chlorid, put in the bottle, and shake at intervals for six or eight hours; let settle over night, then filter off the liquid into a second bottle. The first bottle is then ready for preparation of a fresh supply when needed.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion containing 0.5 grams is evaporated with 10 cc. of the platinum solution and a few drops hydrochloric acid and washed as before.

(3) *Sulphate of potash, kainit, etc.*—In the analysis of kainit, dissolve 10 grams of the pulverized salt in 300 cc. of boiling water, add ammonia to slight excess, then a sufficient quantity of ammonium oxalate to throw down all lime present; cool and make up to 500 cc., mix thoroughly, and filter on a dry filter; take 25 cc., corresponding to 0.5 grams, and proceed to remove the ammonia, as in the analysis of superphosphates; dissolve the residue in hot water plus a few drops of hydrochloric acid and add 15 cc. of platinum solution. In the analysis of high-grade sulphate and of double-manure salt (potassium sulphate, magnesium sulphate, containing about 27 per cent of potassium oxid), make up the solution as above, but omit the precipitation, evaporation, etc.; take an aliquot part equal to 0.5 gram and add 15 cc. of platinum solution. In all cases special care must be taken in the washing with alcohol to remove all the double platinum-sodium chlorid if present. The washing should be continued some time after the filtrate is colorless. Twenty-five cc. of the ammonium chlorid solution are employed instead of 10 cc., and the 25 cc. poured through at least 6 times to remove all sulphates and chlorids. Wash finally with alcohol; dry and weigh as usual.

(4) *Organic compounds.*—In case the potash is contained in organic compounds like tobacco stems, cotton-seed hulls, etc., weigh out 10 grams, saturate with strong sulphuric acid, and ignite in a muffle to destroy organic matter. Add a little strong hydrochloric acid to moisten the mass and warm slightly so as to loosen it in the dish. Proceed then as in (1).

ALTERNATE METHOD FOR POTASH.

Take 10 grams of the prepared sample, boil for 10 minutes with 200 cc. of water, and, after cooling and without filtering, make up to 1,000 cc. and filter through a dry filter. If the sample has 10 to 15 per cent of potassium oxid (kainit), take 50 cc. of the filtrate; if from 2 to 3 per cent of potassium oxid (ordinary potash fertilizers), take 100 cc. of the filtrate. In each case make the volume up to 150 cc., heat to 100°, and add, drop by drop with constant stirring, slight excess of barium chlorid, without filtering; in the same manner add barium hydrate in slight excess. Filter while hot and wash until the precipitate is free of chlorids. Add to the filtrate 1 cc. of strong ammonium hydrate, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat: add now, in fine powder, 0.5 grams pure oxalic acid or 0.75 grams of ammonium oxalate. Filter; wash free of chlorids, evaporate the filtrate to dryness in a platinum dish, and ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cc. or more. To this filtrate, after adding 2 drops of strong hydrochloric acid, are added, in a porcelain dish, 5 to 10 cc. of a solution of 10 grams of platinic chlorid in 100 cc. of water. The mixture is now evaporated on a water bath to a thick sirup, as above, treated with 80 per cent alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cc. of ether, dried for 30 minutes at 100°, and weighed.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed, according to the previous method, with 10 cc. of the half-concentrated solution of ammonium chlorid, which has been saturated by shaking with potassium platinic chlorid.

FACTORS.

The use of the factors 0.3056 for converting potassium platinic chlorid to potassium chlorid and 0.19308 for converting to potassium oxid is continued.

METHODS OF SUGAR ANALYSIS.[1]

DETERMINATION OF WATER.

In sugar or massecuite.—Dry from 2 to 5 grams in a flat dish (nickel or platinum) at the temperature of boiling water for ten hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there is only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not greater than 0.20 per cent.

In honey or molasses.—Proceed as above, using only 1 to 2 grams of substance.

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[1] By the term "sugar analysis" it is understood that the analyses of all the common saccharine substances, viz, juices, sugar, molasses, sirup, starch sugar, and honey, are embraced. In the following directions an attempt will be made to indicate the general principles which should guide the practice of sugar analysis, together with sufficient detail to enable these analyses to be carried out with accuracy and uniformity.

The principal components to be determined in the substances mentioned above are:

1. Water.	4. Nitrogenous bodies.	7. Lactose.
2. Density and total solids.	5. Reducing sugar.	8. Starch sugar.
3. Ash.	6. Sucrose.	9. Raffinose.

DENSITY AND TOTAL SOLIDS.

The density of juices, sirups, etc., is most conveniently determined by means of Baumé's or Brix's hydrometer or areometer, preferably with the latter, as the graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths. It is therefore desirable, for accuracy, that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be as nearly as possible of the same temperature as the air at the time of reading, and if the variation from the standard temperature of the graduation of the spindle amounts to more than 1°, compensation therefor must be made by reference to tables of corrections for temperature.

Care should be observed in taking the density of a juice to allow it to stand in the cylinder until all air bubbles have escaped, before reading the spindles. When it is desired to accurately determine the specific gravity of the sample, a specific-gravity bottle or picnometer is employed.

The per cent of total solids can be approximately ascertained directly from the reading of the Brix hydrometer, or, knowing the reading of the Baumé spindle or the specific gravity of the sample, by reference to the table on page 214, bulletin 31.

DETERMINATION OF ASH.

(a) Heat 5 or 10 grams of the material (sugar, molasses, honey) [1] in a platinum dish[1] of 50 to 100 cc. capacity at 100° until the water is expelled, and then slowly over a flame until intumescence ceases. The dish is then placed in a muffle and heated at low redness until a white ash is obtained.

For soluble ash digest the ash with water, filter through a Gooch crucible, wash with hot water, dry the residue at 100°, and weigh. The difference of weights equals soluble ash.

(b) Use 50 milligrams of zinc oxid to 25 grams of molasses or 50 grams of sugar. Incorporate thoroughly by adding dilute alcohol and mixing; dry and ignite as above. Deduct the weight of zinc used from the weight of ash.

(c) Carbonize the mass at a low heat, dissolve the soluble salts with hot water, burn the residual mass as above, add the solution of soluble salts, and evaporate to dryness at 100°; ignite gently, cool in a desiccator, and weigh.

(d) Saturate the sample with H_2SO_4, dry, ignite gently, then burn in a muffle at low redness. Deduct one-tenth of the weight of the ash, then calculate the per cent.

(e) Dissolve 10 grams of sugar in hot water and filter to remove sand, etc., evaporate the filtrate and washings to dryness; carefully carbonize and extract with hot water until there is no longer any reaction for chlorin. Dry and ignite the residue; weigh as insoluble ash. Add the soluble portion, treat with HCl in slight excess, and evaporate to dryness. Heat until all free HCl is driven off; take up with water and a little HCl, filter, and wash. The residue is silica. Add NH_4OH to the filtrate, boil and filter, wash the residue, which is iron oxid and alumina. To the filtrate add $(NH_4)_2C_2O_4$; evaporate to dryness; ignite and moisten with $(NH_4)_2CO_3$ and re-ignite; dissolve in water, filter, and wash. The residue on the filter represents calcium and magnesium carbonate. Evaporate the filtrate to a small bulk, add $(NH_4)_2CO_3$, and evaporate to dryness; drive off the excess of ammonia cautiously and weigh. This gives the alkalis as carbonates, and this weight, added to the insoluble ash, formerly determined, gives the total carbonate ash.

(f) Thoroughly mix 5 grams of the material with a somewhat larger weight of pure quartz sand in a platinum dish; ignite in a muffle at a moderate red heat.

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[1] If the substance contains tin or any other metal capable of uniting with platinum, a dish made of some other material must be used.

To avoid the correction of one-tenth, as proposed by Scheibler, and one-fifth as proposed by Girard and Violette, when sugars are burned with H_2SO_4, Boyer suggests incineration with benzoic acid, as giving the real quantity of mineral matter without correction.

The benzoic acid is dissolved in alcohol of 90 per cent, 25 grams of the acid to 100 cc. of alcohol; 5 grams of the sugar are weighed in a capsule and moistened with 1 cc. of water. The capsule is heated slowly in order to caramelize the sugar without carbonizing it; 2 cc. of the benzoic-acid solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then raised until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors, which render the mass extremely porous, especially if a circular motion is imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized.

The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Ammonium benzoate may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonia salt leaves a residue on incineration. In addition to giving the mineral matters directly, this method permits the determination of their composition also, a matter of no small importance.

DETERMINATION OF NITROGEN.

Any of the methods adopted by the association for the estimation of nitrogen may be used.

REDUCING SUGARS.

Reagents (Soxhlet's modification of Fehling's solution).

(1). 34. 639 grams of $CuSO_4$, $5H_2O$, dissolved and diluted to 500 cc. with water; (2). 173 grams of Rochelle salt, 50 grams of NaOH, dissolved and diluted to 500 cc. with water. Mix equal volumes of these solutions immediately before use. This mixture is the "mixed copper reagent" to be used for all the methods given below except Allihn's method for dextrose, for which a special reagent must be used.

(a) *Approximate volumetric method for rapid work.*—Place 10 cc. of the mixed copper reagent in a large test tube and add 10 cc. of distilled water. Heat to boiling, and gradually add small portions of the solution of the material to be tested, boiling to complete the reaction after each addition, until the copper has been completely precipitated. Two minutes boiling is required for complete precipitation when the full amount of sugar solution has been added in one portion. When the end reaction is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, a small portion of the liquid is removed by means of Knorr's modification of Wiley's filtering tube (any other rapid means of filtration may be used that removes but a small portion of the hot liquid), is transferred to a small porcelain crucible or test plate, acidified with dilute acetic acid, and tested for copper with a dilute solution of potassium ferrocyanid. The sugar solution should be of such a strength as will give a burette reading of 15–20 cc., and the number of successive additions should be as small as possible.

Since the factor for calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of a pure sample of the sugar that he desires to determine and keeping the conditions the same as those used for the determinations. For the standardization of a solution for the

determination of invert sugar in sugar-house products, dissolve 2.5 grams of pure sucrose in 100 cc. of water, add 10 cc. of hydrochloric acid (specific gravity 1.188), and invert according to the method given below for double polarization. Neutralize the acid with sodium carbonate and dilute to 1 liter. The 2.5 grams of sucrose become 2.6316 grams of invert sugar. The weight of invert sugar equivalent to 10 cc. of the copper reagent is calculated as follows:

$$\frac{2.6316 \times \text{ by number of cc. of the standard sugar solution used}}{1000} = X,$$

the weight of invert sugar required to completely precipitate the copper in 10 cc. of the reagent under the conditions used for the test titration. For the calculation of the result of the titration of an unknown solution:

Let X = the factor obtained as above;

    V = the number of cc. of unknown sugar solution required to precipitate the copper from 10 cc. of copper solution;

    W = the weight of the material under examination in 1 cc. of the solution.

Then $\dfrac{100\,X}{V\,W}$ = per cent of invert sugar in the sample.

The calculation can be much simplified by so standardizing the copper reagent that 50 milligrams of invert sugar will be required to reduce the copper from 10 cc. of the copper reagent. The various tables given in works on sugar analysis then become applicable. These tables are arranged for a "glucose normal solution" containing 5 grams of the material to be examined in 100 cc. When the weight per 100 cc. is more or less than 5 grams the number found in the table is increased or diminished accordingly.

(b) *Soxhlet's exact volumetric method.*—A preliminary titration is made to determine the approximate percentage of reducing sugar in the material under examination. A solution is then prepared which contains approximately 1 per cent of reducing sugar. Place in a beaker 100 cc. of the mixed copper reagent and approximately the amount of the sugar solution for its complete reduction. Boil for two minutes. Filter through a plaited filter and test a portion of the filtrate for copper by use of acetic acid and potassium ferrocyanid. Repeat the test, varying the volume of sugar solution, until two successive amounts of sugar solution are found, which differ by 0.1 cc., one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these two readings is taken as the volume of the solution required for the complete precipitation of 100 cc. of the copper reagent.

Under these conditions 100 cc. of the mixed copper reagent require 0.475 gram of anhydrous dextrose, or 0.494 gram of invert sugar for complete reduction. The percentage is calculated by the following formula:

    W = the weight of the sample in 1 cc. of the sugar solution;

    V = the volume of the sugar solution required for the complete reduction of 100 cc. of the copper reagent.

Then $\dfrac{100 \times 0.475}{V\,W}$ = per cent of dextrose,

or $\dfrac{100 \times 0.494}{V\,W}$ = per cent of invert sugar.

(c) *Gravimetric method for materials containing 1 per cent or less of invert sugar.*— The solution of the material to be examined is so prepared as to contain 20 grams in 100 cc., and it must be freed from suspended impurities by filtration through paper or by use of lead acetate, removing the excess of lead by means of sodium carbonate. In a beaker of 250 cc. capacity place 50 cc. of the mixed copper reagent and 50 cc. of the sugar solution. Heat this mixture at such a rate that approximately four minutes are required to bring it to the boiling point and boil for exactly two minutes. Add 100 cc. of cold, recently boiled, distilled water. Filter immediately through a

weighed filtering tube of hard glass under pressure. The asbestos film in the filtering tube is supported by a perforated disk or cone of platinum and should be washed free from loose fibers before weighing and moistened previous to the filtration. The tube is provided with a detachable funnel during the filtration so that none of the precipitate accumulates near the top, where it could be removed by the cork used during the reduction of the cuprous oxid. The precipitate is all transferred to the filter and thoroughly washed with hot water, following the water by alcohol and ether successively. After being dried the tube is connected with an apparatus for supplying a continuous current of dry hydrogen, gently heated until the cuprous oxid is completely reduced to the metallic state, cooled in the current of hydrogen, and weighed. The increase in weight is the weight of copper reduced by 10 grams of the sample. The corresponding percentage of invert sugar is found by use of the following table:

Herzfeld's table for the determination of invert sugar in materials containing 1 per cent or less of invert sugar.[1]

Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.
Milligrams.	*Per cent.*	*Milligrams.*	*Per cent.*	*Milligrams.*	*Per cent.*
50	0.05	120	0.40	190	0.79
55	0.07	125	0.43	195	0.82
60	0.09	130	0.45	200	0.85
65	0.11	135	0.48	205	0.88
70	0.14	140	0.51	210	0.90
75	0.16	145	0.53	215	0.93
80	0.19	150	0.56	220	0.96
85	0.21	155	0.59	225	0.99
90	0.24	160	0.62	230	1.02
95	0.27	165	0.65	235	1.05
100	0.30	170	0.68	240	1.07
105	0.32	175	0.71	245	1.10
110	0.35	180	0.74		
115	0.38	185	0.76		

[1] Zeitschrift des Vereins für die Rübenzucker-Industrie des Deutschen Reichs, 1885, **35**, 967.

The weight of copper may be obtained as follows: The filtration after reduction is made in a Gooch crucible, and the beaker and precipitate thoroughly washed with hot water without any effort to transfer the precipitate to the filter. Wash the asbestos film and the adhering cuprous oxid into the beaker by means of a wash bottle containing hot dilute nitric acid. After the copper is all in solution refilter through a Gooch crucible with a thin film of asbestos and wash thoroughly with hot water. Add 10 cc. of dilute sulphuric acid (containing 200 cc. of sulphuric acid—specific gravity 1.84—per liter) and evaporate the filtrate on the steam bath until the copper salt has largely crystallized out. Heat carefully on a hot plate or over a piece of asbestos board until the evolution of white fumes shows that the excess of nitric acid is removed. Add 8 to 10 drops of nitric acid (specific gravity 1.42) and rinse into a platinum dish of 100 to 125 cc. capacity. Precipitate the copper by electrolysis. Wash with water thoroughly before breaking the current, remove the dish from the circuit, wash with alcohol and ether successively, dry at a temperature that can be borne by the hand, and weigh. The evaporation of the liquid for the removal of the excess of nitric acid may be avoided by the following process: Transfer the asbestos film from the crucible to the beaker by means of a glass rod and rinse the crucible with about 30 cc. of a boiling mixture of dilute nitric and sulphuric acids, containing 65 cc. of sulphuric acid (specific gravity 1.84) and 50 cc. of nitric acid

(specific gravity 1.42) per liter; or the asbestos film with the adhering cuprous oxid may be treated by the method of Formanek given below, by transferring to the beaker containing the rest of the suboxid and washing the crucible with 2–4 cc. of nitric acid (strong) to remove all adhering precipitate, receiving the solutions in the beaker. The crucible is then washed with 5–10 cc. water which is also received in the beaker. After all the cuprous oxid in the beaker is dissolved the crucible is worked with an additional quantity of water. Refilter through a Gooch crucible and wash thoroughly with hot water, limiting the volume of the filtrate to 75 to 80 cc. Transfer to a platinum dish and electrolyze as described above. Wash before interrupting the current, using water acidulated with sulphuric acid at first.

(d) *Gravimetric method for materials containing more than 1 per cent of invert sugar.*—Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc. after clarification and the removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, 5, etc., cc. of this solution to each successively. Add 5 cc. of the mixed copper reagent to each, heat to boiling, boil two minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place twenty times this volume of the sugar solution in a 100 cc. flask, dilute to the mark, and mix well. Use 50 cc. of the solution for the determination, which is conducted as described under c, until the weight of copper is obtained. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;

P = the polarization of the sample;

W = the weight of the sample in the 50 cc. of the solution used for determination;

F = the factor obtained from the table for conversion of copper to invert sugar;

$\dfrac{Cu}{2}$ = approximate absolute weight of invert sugar = Z;

$Z \times \dfrac{100}{W}$ = approximate per cent of invert sugar = y;

$\dfrac{100\,P}{P+y}$ = R, relative number for sucrose;

$100 - R = I$, relative number for invert sugar;

$\dfrac{Cu\,F}{W}$ = per cent of invert sugar.

Z facilitates reading the vertical columns; and the ratio of R to I, the horizontal columns of the table, for the purpose of finding the factor (F) for calculation of copper to invert sugar.

Example: The polarization of a sugar is 86.4, and 3.256 grams of it (W) are equivalent to 0.290 grams of copper. Then:

$$\frac{Cu}{2} = \frac{.290}{2} = 0.145 = Z$$

$$Z \times \frac{100}{W} = 0.145 \times \frac{100}{3.256} = 4.45 = Y$$

$$\frac{100\,P}{P+y} = \frac{8640}{86.4 + 4.45} = 95.1 = R$$

$$100 - R = 100 - 95.1 = I = 4.9$$

$$R : I = 95.1 : 4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet we find the factor 51.2, which enters into the final calculation:

$$\frac{Cu\,F}{W} = \frac{0.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert sugar.}$$

Meissl and Hiller's factors for the determination of more than 1 per cent of invert sugar.[1]

Ratio of sucrose to invert sugar = R : I.	Approximate absolute weight of invert sugar = Z.						
	200 milli-grams.	175 milli-grams.	150 milli-grams.	125 milli-grams.	100 milli-grams.	75 milli-grams.	50 milli-grams.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
0 : 100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	.52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

[1] Zeitschrift des Vereins für der Rübenzucker-Industrie des Deutschen Reichs, 1889, **39**, 735.

(c) *Allihn's gravimetric method for the determination of dextrose.*

Reagents:

I. 34.639 grams of $CuSO_4, 5H_2O$, dissolved in water and diluted to 500 cc.

II. 173 grams of Rochelle salts } dissolved in water and diluted to 500 cc.
125 grams of KOH,

Place 30 cc. of the copper solution (I), 30 cc. of the alkaline tartrate solution (II), and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the solution of the material to be examined, which must be so prepared as not to contain more than 1 per cent of dextrose, and boil for two minutes. Filter immediately without diluting and obtain the weight of copper by one of the methods given under (c). The corresponding weight of dextrose is found by the following table:

Allihn's table for the determination of dextrose.[1]

Milli-grams of cop-per.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of dex-trose.
10	6.1	22	12.0	34	18.0	46	23.9	58	29.8
11	6.6	23	12.5	35	18.5	47	24.4	59	30.3
12	7.1	24	13.0	36	18.9	48	24.9	60	30.8
13	7.6	25	13.5	37	19.4	49	25.4	61	31.3
14	8.1	26	14.0	38	19.9	50	25.9	62	31.8
15	8.6	27	14.5	39	20.4	51	26.4	63	32.3
16	9.0	28	15.0	40	20.9	52	26.9	64	32.8
17	9.5	29	15.5	41	21.4	53	27.4	65	33.3
18	10.0	30	16.0	42	21.9	54	27.9	66	33.8
19	10.5	31	16.5	43	22.4	55	28.4	67	34.3
20	11.0	32	17.0	44	22.9	56	28.8	68	34.8
21	11.5	33	17.5	45	23.4	57	29.3	69	35.3

[1] J. prakt. Chem., 1880, **22**, 46.

Allihn's table for the determination of dextrose[1]—Continued.

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
70	35.8	122	62.1	174	89.0	226	116.4	278	144.4
71	36.3	123	62.6	175	89.5	227	116.9	279	145.0
72	36.8	124	63.1	176	90.0	228	117.4	280	145.5
73	37.3	125	63.7	177	90.5	229	118.0	281	146.1
74	37.8	126	64.2	178	91.1	230	118.5	282	146.6
75	38.3	127	64.7	179	91.6	231	119.0	283	147.2
76	38.8	128	65.2	180	92.1	232	119.6	284	147.7
77	39.3	129	65.7	181	92.6	233	120.1	285	148.3
78	39.8	130	66.2	182	93.1	234	120.7	286	148.8
79	40.3	131	66.7	183	93.7	235	121.2	287	149.4
80	40.8	132	67.2	184	94.2	236	121.7	288	149.9
81	41.3	133	67.7	185	94.7	237	122.3	289	150.5
82	41.8	134	68.2	186	95.2	238	122.8	290	151.0
83	42.3	135	68.8	187	95.7	239	123.4	291	151.6
84	42.8	136	69.3	188	96.3	240	123.9	292	152.1
85	43.4	137	69.8	189	96.8	241	124.4	293	152.7
86	43.9	138	70.3	190	97.3	242	125.0	294	153.2
87	44.4	139	70.8	191	97.8	243	125.5	295	153.8
88	44.9	140	71.3	192	98.4	244	126.0	296	154.3
89	45.4	141	71.8	193	98.9	245	126.6	297	154.9
90	45.9	142	72.3	194	99.4	246	127.1	298	155.4
91	46.4	143	72.9	195	100.0	247	127.6	299	156.0
92	46.9	144	73.4	196	100.5	248	128.1	300	156.5
93	47.4	145	73.9	197	101.0	249	128.7	301	157.1
94	47.9	146	74.4	198	101.5	250	129.2	302	157.6
95	48.4	147	74.9	199	102.0	251	129.7	303	158.2
96	48.9	148	75.5	200	102.6	252	130.3	304	158.7
97	49.4	149	76.0	201	103.1	253	130.8	305	159.3
98	49.9	150	76.5	202	103.7	254	131.4	306	159.8
99	50.4	151	77.0	203	104.2	255	131.9	307	160.4
100	50.9	152	77.5	204	104.7	256	132.4	308	160.9
101	51.4	153	78.1	205	105.3	257	133.0	309	161.5
102	51.9	154	78.6	206	105.8	258	133.5	310	162.0
103	52.4	155	79.1	207	106.3	259	134.1	311	162.6
104	52.9	156	79.6	208	106.8	260	134.6	312	163.1
105	53.5	157	80.1	209	107.4	261	135.1	313	163.7
106	54.0	158	80.7	210	107.9	262	135.7	314	164.2
107	54.5	159	81.2	211	108.4	263	136.2	315	164.8
108	55.0	160	81.7	212	109.0	264	136.8	316	165.3
109	55.5	161	82.2	213	109.5	265	137.3	317	165.9
110	56.0	162	82.7	214	110.0	266	137.8	318	166.4
111	56.5	163	83.3	215	110.6	267	138.4	319	167.0
112	57.0	164	83.8	216	111.1	268	138.9	320	167.5
113	57.5	165	84.3	217	111.6	269	139.5	321	168.1
114	58.0	166	84.8	218	112.1	270	140.0	322	168.6
115	58.6	167	85.3	219	112.7	271	140.6	323	169.2
116	59.1	168	85.9	220	113.2	272	141.1	324	169.7
117	59.6	169	86.4	221	113.7	273	141.7	325	170.3
118	60.1	170	86.9	222	114.3	274	142.2	326	170.9
119	60.6	171	87.4	223	114.8	275	142.8	327	171.4
120	61.1	172	87.9	224	115.3	276	143.3	328	172.0
121	61.6	173	88.5	225	115.9	277	143.9	329	172.5

[1] J. prakt. Chem., 1880, 22, 46.

Allihn's table for the determination of dextrose [1]—Continued.

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
330	173.1	357	188.3	384	203.7	411	219.3	438	235.1
331	173.7	358	188.9	385	204.3	412	219.9	439	235.7
332	174.2	359	189.4	386	204.8	413	220.4	440	236.3
333	174.8	360	190.0	387	205.4	414	221.0	441	236.9
334	175.3	361	190.6	388	206.0	415	221.6	442	237.5
335	175.9	362	191.1	389	206.5	416	222.2	443	238.1
336	176.5	363	191.7	390	207.1	417	222.8	444	238.7
337	177.0	364	192.3	391	207.7	418	223.3	445	239.3
338	177.6	365	192.9	392	208.3	419	223.9	446	239.8
339	178.1	366	193.4	393	208.8	420	224.5	447	240.4
340	178.7	367	194.0	394	209.4	421	225.1	448	241.0
341	179.3	368	194.6	395	210.0	422	225.7	449	241.6
342	179.8	369	195.1	396	210.6	423	226.3	450	442.2
343	180.4	370	195.7	397	211.2	424	226.9	451	242.8
344	180.9	371	196.3	398	211.7	425	227.5	452	243.4
345	181.5	372	196.8	399	412.3	426	228.0	453	244.0
346	182.1	373	197.4	400	212.9	427	228.6	454	244.6
347	182.6	374	198.0	401	213.5	428	229.2	455	245.2
348	183.2	375	198.6	402	214.1	429	229.8	456	245.7
349	183.7	376	199.1	403	214.6	430	230.4	457	246.3
350	184.3	377	199.7	404	215.2	431	231.0	458	246.9
351	184.9	378	200.3	405	215.8	432	231.6	459	247.5
352	185.4	379	200.8	406	216.4	433	232.2	460	248.1
353	186.0	380	201.4	407	217.0	434	232.8	461	248.7
354	186.6	381	202.0	408	217.5	435	233.4	462	249.3
355	187.2	382	202.5	409	218.1	436	233.9	463	249.9
356	187.7	383	203.1	410	218.7	437	234.5		

[1] J. prakt. Chem., 1880, 22, 46.

(*f*) *Modified method of Formanek for electrolytic estimation of reduced copper.*—To the solution obtained by the treatment of the precipitated suboxid with nitric acid and hot water add a sufficient quantity of water to reduce the nitric acid content of the solution below 5 per cent, and electrolyze.

Instead of dissolving the suboxid with nitric acid on the filter, the asbestos filter and contents are transferred from the funnel to the beaker used in the precipitation, and the funnel is washed out with a 4 per cent nitric-acid solution. This acid solution is added to the contents of the beaker until a bulk of about 200 cc. is attained.

Two platinum cylinders are then immersed in the liquid and connections are made with a battery, the larger cylinder being the cathode.

ESTIMATION OF SUCROSE.

(*a*) *In sugars, massecuites, etc.*—Take the normal weight in tared dish for the instrument employed; wash into a 100 cc. flask; add water until volume is 80 to 85 cc. When the crystals are all dissolved add sufficient lead acetate to throw down all precipitable matter. With molasses and massecuites add sufficient acetic acid to convert the sub into the neutral acetate. Make up to mark, using a little ether spray to dissolve bubbles; filter, throwing away the first 10 to 15 cc.; place in observation tube and polarize. If too dark to read filter through finely powdered dry bone black, rejecting the first 30 to 40 cc.

For adjusting the polariscope, graduating flasks, etc., the method of the United

States Bureau of Internal Revenue, adopted last year, was continued. (See Bull. 31, pp. 228–232.)

(*b*) *In juices, etc.*—Transfer, by means of a pipette, to the tared sugar dish the normal, or multiple normal, weight of the juice or sirup to be analyzed.

In the case of juices and thin sirups, the contents of the dish are at once washed into the 100 cc. flask.

The solution in the flask is made up to 80 or 90 cc., and the least amount of basic lead-acetate solution sufficient to decolorize the contents, is added. The solution is then made up to the 100 cc. mark and thoroughly shaken before filtering. In case much foaming and bubbling takes place, rendering gauging difficult, the addition of a few drops of ether is found advantageous. The solution is then poured upon a dry filter and the filtrate is collected in a dry beaker, the first portions to pass through being rejected. If it is found impossible to obtain a perfectly clear filtrate it may be necessary to decolorize with dry powdered bone black, either mixed with the liquid itself or placed upon the filter.

In case of dark-colored products, the addition of alumina cream immediately after the lead subacetate is quite advantageous, the quantity of the former employed being in excess of that of the latter. For many sugars the use of alumina cream alone will produce a satisfactory clarification. Where double polarization is employed, acetic acid should be added to the filtrate used for the direct reading, in sufficient proportion to break up the compound of lead and levulose which has been formed.

Preparation of lead subacetate solution.—Boil an aqueous solution of lead acetate with an excess of lead oxid (PbO) for half an hour. and make filtered solution of a concentration of not less than 1.25 specific gravity. Solid subacetate of lead may be substituted for the normal salt and oxide in the preparation of the solution.

Alumina cream.—Prepare a cold saturated solution of alum in water and divide into two unequal portions. Add a slight excess of ammonium hydrate to the larger portion, and then add by degrees the remaining alum solution until a faintly acid reaction is secured.

Optical method by inversion.—For raw sugar, molasses, etc.

(*a*) *Method of Clerget.*—Make up the solution as above, and place 50 cc. of the filtrate in a flask marked at 50 and 55 cc. Fill to the upper mark with pure fuming HCl and mix well; place in water and heat until the thermometer, with the bulb as near the center of the sugar solution in the flask as possible, marks 68°, consuming about ten minutes in the heating; remove, cool quickly to room temperature, and polarize, noting the temperature. If the sample contained originally any invert sugar the second polarization should be made at approximately the same temperature as the first. The percentage of sucrose is then calculated by the following formula:

$$S = \text{percentage of sucrose.}$$
$$a = \text{first polarization.}$$
$$b = \text{second polarization (usually to the left).}$$
$$a \pm b = \text{sum of the polarizations.}$$
$$t = \text{temperature of observation.}$$

Then
$$S = \frac{a \pm b}{144 - \frac{t}{2}}$$

a and *b* are to be added when of different signs and the difference taken when of the same sign.

(*b*) *The official German method* may be used as follows: 26.048 grams are dissolved in a sugar flask and the solution made up to 100 cc.; 50 cc. of this solution are transferred by means of a pipette to a 50 or 55 cc. flask, cleared, and polarized, the reading being corrected for the extra 5 cc. The liquid adhering to the pipette is washed into the 100 cc. flask containing the remaining 50 cc. (13.024 grams), 5 cc. of concen-

trated hydrochloric acid (38 per cent, specific gravity 1.18 at 15°) added and the flask heated for fifteen minutes at 67° to 70° in a water bath. The temperature should not exceed this limit. The flask is then cooled rapidly and the solution made up to 100 cc. If the liquid is colored it is shaken with $\frac{1}{2}$ to 1 gram of bone black and filtered through a double filter. It is then polarized in a 200 mm. tube which is provided with a thermometer. As the rotatory power of invert sugar is much influenced by the temperature, this factor must be regarded. The reading is best taken between 18° and 22° and the temperature accurately determined. The reading must also be doubled on account of the dilution of the liquid.

To calculate per cent of sucrose (R) the two readings are added together, the sum (S) multiplied by 100 and divided by $142.4 - \frac{1}{2}t$ where t is the temperature at which the invert reading was taken. If the temperature is exactly 20° the result can be made more accurate by substituting 142.66 for 142.4; thus:

$$R = \frac{100\ S}{142.66\ \frac{20}{2}} = \frac{100\ S}{132.66} = 0.7538\ S.$$

If very much invert sugar is present, both the direct and indirect readings must be taken at the same temperature.

(c) *Method of Lindet.*—Place 50 cc. of the filtrate as above in a flask marked at 100.5 cc.; add 5 grams of powdered zinc (zinc dust), place in boiling water, and when at the boiling point add, little by little, 5 cc. of strongest HCl. After the acid has all been added, cool and make up to mark. Polarize in a 400-mm. tube, or multiply the reading in a 200-mm. tube by 2. Calculate the percentage of sucrose as before.

(d) *Gravimetric method.*—Determine first any reducing sugar in the sample; then invert sucrose, neutralize free acid, and redetermine the reducing sugar. Deduct the percentage of reducing sugar obtained at first, and the remainder will be reducing sugar derived from sucrose; multiply this number by .95 to obtain the percentage of sucrose in the sample.

ELECTIVE METHODS.

The elective methods remain the same as in Bulletin 28, pp. 217-228.

METHODS OF ANALYSIS OF FOODS AND FEEDING STUFFS.

PREPARATION OF SAMPLE.

The substance is to be ground and passed through a sieve with circular holes 1 millimeter in diameter.

DETERMINATION OF MOISTURE.

Dry from 2 to 3 grams of the substance for five hours, at the temperature of boiling water, by passing a current of dry hydrogen through the sample. If the substance be held in a glass vessel the latter should not be in contact with the boiling water.

DETERMINATION OF ASH.

Char 2 to 3 grams of the substance and burn to whiteness at the lowest possible red heat. If a white ash can not be obtained in this manner, exhaust the charred mass with water; collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the above aqueous extract, and heat the whole to a low redness till the ash is white.

DETERMINATION OF ETHER EXTRACT.

Extract 2 to 3 grams of the substance dried as for the determination of the moisture, with anhydrous and alcohol-free ether, sixteen hours. Dry the extract, by exposure to the full heat of boiling water, to constant weight.

ALTERNATE METHOD FOR ETHER EXTRACT.

In determining hygroscopic water, as above, continue the drying until the loss of weight in 30 minutes is reduced to 1 milligram or less; extract the dried substance for sixteen hours as directed, dry again, and give loss of weight as ether extract.

Anhydrous ether.—To prepare the anhydrous alcohol-free ether required for estimation of fat, take any of the commercial brands of ether, wash with two or three successive portions of distilled water, add sticks of solid caustic soda or potash until most of the water has been abstracted from the ether. Carefully cleaned metallic sodium, cut into small pieces, is now added until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered in order to allow any accumulating hydrogen gas to escape; and it may be drawn off with a pipette as required.

CRUDE PROTEIN.

Determine nitrogen as directed for nitrogen in fertilizers and multiply the result by 6.25 for the crude protein.

DETERMINATION OF ALBUMINOID NITROGEN—STUTZER'S METHOD.

To 0.7 to 0.8 gram of the substance add 100 cc. of water in a beaker, heat to boiling, or in the case of substances rich in starch heat on the water bath ten minutes, add a quantity of cupric hydrate mixture containing 0.5 to 0.6 gram of the hydrate stir thoroughly, filter when cold, wash with cold water, and put the filter and its contents into the concentrated sulphuric acid for the determination of nitrogen. The filter papers used must be practically free of nitrogen. Add sufficient potassium-sulphid solution to completely precipitate all copper and mercury. If the substance examined consist of seed of any kind, or residues of seeds, such as oil cake or anything else rich in alkaline phosphates, add a few cc. of a concentrated solution of alum just before adding the cupric hydrate, and mix well by stirring. This serves to decompose the alkaline phosphates. If this is not done cupric phosphate and free alkali may be formed, and the protein-copper may be partially dissolved in the alkaline liquid.

Cupric hydrate.—Prepare cupric hydrate as follows: Dissolve 100 grams of pure cupric sulphate in 5 liters of water, and add 25 cc. of glycerol; add dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cc. of glycerol per liter, and then wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerol, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture.

DETERMINATION OF CRUDE FIBER.

(1) In feeding stuffs poor in starch.—Extract 2 grams of the substance with ordinary ether, at least nearly completely, or take the residue from the determination of the ether extract. To this residue, in a 500 cc. flask, add 200 cc. of boiling 1.25 per cent sulphuric acid; connect the flask with a return-flow condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid. Filter; wash thoroughly with boiling water till the washings are no longer acid; rinse the substance back into the same flask with 200 cc. of a boiling 1.25 per cent solution of sodium hydrate at least almost free from sodium carbonate; boil at once, and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter in a Gooch crucible, and wash with boiling water till the washings are neutral; dry at 110°; weigh; incinerate completely, and give the loss of weight for crude fiber.

The filter used for the first filtration may be linen, one of the forms of glass-wool or asbestos filters recommended in the last report, or any other form that secures clear and reasonably rapid filtration. The solutions of sulphuric acid and sodium hydrate are to be made up of the specified strength accurately by titration, and not merely from specific gravity.

(2) *In feeding stuffs rich in starch.*—Proceed as under (1) using 2.5 per cent acid and alkali in place of the solution of 1.25 per cent strength.

METHODS OF ANALYSIS OF DAIRY PRODUCTS.

BUTTER.

MICROSCOPIC EXAMINATION.

Place a small portion of the fresh samples, taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc. Examine the same specimen with polarized light and a selenite plate without the use of oil. Pure fresh butter will neither show crystals nor a particolored field with selenite. Other fats melted and cooled and mixed with butter will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve 4 or 5 cc. of the fat in 15 cc. of ether in a test tube. Close the tube with a loose plug of cotton wool and allow to stand 12 to 24 hours at room temperature (20° to 25°). When crystals form at the bottom of the tube they are removed with a pipette, glass rod, or tube, placed on a slide, covered, and examined. The crystals formed by later deposits may be examined in a similar way.

SAMPLING.

If large quantities of butter are to be sampled a butter trier or sampler may be used. The portions thus drawn, about 500 grams, are to be perfectly melted in a closed vessel at as low a heat as possible, and when melted the whole is to be shaken violently for some minutes till the mass is homogeneous.[1] A portion is then poured into the vessel from which it is to be weighed for analysis, and should nearly or quite fill it. This sample should be kept in a cold place till analyzed.

DETERMINATION OF WATER.

One and five-tenths to 2.5 grams are dried to constant weight at the temperature of boiling water in a dish with flat bottom, having a surface of at least 20 cm².

The use of clean dry sand or asbestos with the butter is admissible, and is necessary if a dish with round bottom is employed.

DETERMINATION OF FAT.

The dry butter from the water determination is dissolved in the dish with absolute ether, or with 76° benzine. The contents of the dish are then transferred to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent, and are washed till free from fat. The crucible and contents are heated at the temperature of boiling water till the weight is constant. The weight of fat is calculated from the data obtained.

ALTERNATE METHOD FOR FAT.

Water may be determined by drying the butter on asbestos or sand, and the fat extracted by anhydrous alcohol-free ether. The extract, after evaporation of the ether, is heated to constant weight at the temperature of boiling water and weighed.

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[1] The mass must be sufficiently solidified to prevent the separation of the water and fat after it is placed in the bottle.

DETERMINATION OF CASEIN AND ASH.

The crucible containing the residue from the fat determination, consisting of casein and ash, is covered and heated, gently at first, gradually raising the temperature to just below redness. The cover may then be removed and the heat continued till the contents of the crucible are white. The loss in weight of the crucible and contents represents casein, and the residue in the crucible mineral matter. In this mineral matter, dissolved in water slightly acidulated with nitric acid, chlorin may be determined gravimetrically with silver nitrate, or volumetrically, using potassium chromate as an indicator.

DETERMINATION OF SALT.

The amount of the butter or butter substitute to be taken is from 5 to 10 grams; weigh in a counterpoised beaker-glass. The butter is placed, in portions of about 1 gram at a time, in the beaker, these portions being taken from different parts of the sample. Hot water is now added (about 20 cc.) to the beaker containing the butter, and after it has melted the liquid is poured into the bulb of a separating funnel. The stopper is now inserted and the contents shaken for a few moments. After standing until the fat has all collected on top of the water, the stopcock is opened and the water is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass. Hot water is again added to the beaker, and the foregoing process is repeated from 10 to 15 times, using each time 10 to 20 cc. of water. The resulting washings contain all but a mere trace of the NaCl originally present in the butter. The sodium chlorid is now determined in the filtrate by a standard solution of $AgNO_3$, using a few drops of a saturated solution of potassium chromate as an indicator.

SPECIFIC GRAVITY.

Standardization of flasks.—Use a small specific-gravity flask of from 25 to 30 cc. capacity. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried. After cooling in a desiccator, the weight of the flask and stopper is accurately determined.

The flask is filled with freshly boiled and still hot distilled water, and placed in a bath of pure distilled water. The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper[1] is then inserted, the flask removed, wiped dry, and, after it is nearly cooled to room temperature, placed in the balance, and weighed when balance temperature is reached.

Weight of fat at the temperature of boiling water.—The flask is emptied of its water, rinsed with alcohol and ether, and dried again for a few minutes at the temperature of boiling water. It is then filled with the dry, hot, fresh-filtered fat, which should be be entirely free from air bubbles, and replaced in the water bath, kept for thirty minutes at the temperature of boiling water, removed, and treated as above. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example.

	Grams.
Weight of flask No. 22, dry	10.0197
Weight of flask No. 22, plus water	37.3412
Weight of water	27.3215
Weight of flask No. 22, plus fat	34.6111
Weight of fat	24.5914

Specific gravity = 24.5914 ÷ 27.3215 = .90008.

[1] The stopper should be kept for a few minutes before use in hot distilled water

The weight of the flask dry and empty, and the weight of water at boiling temperature contained therein, may be used constantly if great care be taken in handling and cleaning the apparatus.

Example.

	Grams.
Weight of flask No. 10, dry and empty	10.0028
Weight of flask after three weeks' use	10.0030

Alternate method of standardizing flasks.—Formula for calculating the volume V, in cubic centimeters, of a glass vessel from the weight P of water of temperature t contained therein, and the volume V' at any other temperature t' (from Landolt and Börnstein's physical-chemical tables, p. 39, Table 17):

$$V = P \frac{p}{d}$$

$$V' = P \frac{p}{d} \left[1 + \gamma \, (t' - t) \right]$$

p = weight (in brass weights) of 1 cc. of H_2O in vacuo. (This is so nearly 1 that it will not affect the result in the fifth place of decimals, and may therefore be disregarded.) Hence the formula stands:

$$V' = P \frac{1}{d} \left[1 + \gamma \, (t' - t) \right]$$

d = density of water at temperature t.
γ = .000025, the cubical expansion coefficient of glass.

MELTING POINT.

The apparatus for determining the melting point consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a cathetometer for reading the thermometer (this may be done with an eyeglass if held steadily and properly adjusted); (3) a thermometer; (4) a tall beaker glass 35 cm. high and 10 cm. in diameter; (5) a test tube 30 cm. long and 3.5 cm. in diameter; (6) a stand for supporting the apparatus; (7) some method of stirring the water in the beaker, for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker; (8) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20 cm. on a smooth piece of ice floating in water. The disks thus formed are from 1 to 1.5 cm. in diameter, and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test tube already described until it is nearly half full. The test tube is then nearly filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube, and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker glass is slowly heated, and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2 degrees of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting point of the fat under examination.

The distilled water for floating the piece of ice on which the disks are made should be recently boiled, to free it of all air particles.

The edge of the disks should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place, and the disk adhere to the sides of the tube, a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example.

Melting point of sample of butter: °C.
1st trial 33.15
2d trial 33.05
3d trial 33.00

ESTIMATION OF VOLATILE ACIDS.

Reagents.

(1) *Caustic soda solution.*—100 grams of NaOH are dissolved in 100 cc. of pure water. The caustic soda should be as free as possible from carbonates, and be preserved from contact with the air.

(2) *Alcohol*, of about 95 per cent, redistilled with caustic soda.

(3) *Acid.* Solution of sulphuric acid containing 25 cc. of strongest H_2SO_4 in 1,000 cc. of water.

(4) *Barium hydrate.*—An accurately standardized, approximately decinormal solution of barium hydrate.

(5) *Indicator.*—Alcoholic solution of phenol-phthalein.

Apparatus.

(1) *Saponification flasks* 250 to 300 cc. capacity of hard, well-annealed glass capable of resisting the tension of alcohol vapor at 100°.

(2) *A pipette* graduated to deliver 40 cc.

(3) *Distilling apparatus.*

(4) *Burette.*—An accurately calibrated burette reading to tenths of a cc.

Manipulation.

Weighing the fat.—The butter or fat to be examined should be melted, and kept in a dry warm place at about 60° for two or three hours, until the moisture and curd have entirely settled out. The clean supernatant fat is poured off and filtered through a dry filter paper in a jacketed funnel containing boiling water. Should the filtered fat in a fused state not be perfectly clear, the treatment above mentioned must be repeated.

The saponification flasks are prepared by having them thoroughly washed with water, alcohol, and ether, wiped pefectly dry on the outside, and heated for one hour to boiling temperature. The flasks should then be placed in a tray by the side of the balance and covered with a silk handkerchief until they are perfectly cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed. The weight of the flasks having been accurately determined, they are charged with the melted fat in the following way:

A pipette with a long stem, marked to deliver 5.75 cc. is warmed to a temperature of about 50°. The fat having been poured back and forth once or twice into a dry beaker in order to thoroughly mix it, is taken up in the pipette and the nozzle of the pipette carried to near the bottom of the flask, having been previously wiped to remove any adhering fat, and 5.75 cc. of fat are allowed to flow into the flask. After the flasks have been charged in this way, they should be re-covered with the silk handkerchief and allowed to stand fifteen or twenty minutes, when they are again weighed.

The saponification.—Ten cc. of 95 per cent alcohol are added to the fat in the flask, and then 2 cc. of the concentrated soda solution; a soft cork stopper is now inserted in the flask and tied down with a piece of twine. The saponification is then completed by placing the flask upon the water or steam bath. The saponification, which should last one hour, should be gently rotated from time to time, being careful not to project the soap for any distance up its sides. At the end of an hour the flask, after having been cooled to near the room temperature, is opened.

Removal of the alcohol.—The stoppers having been laid loosely in the mouth of the flask, the alcohol is removed by dipping the flask into a steam bath. The steam should cover the whole of the flask except the neck. After the alcohol is nearly removed, frothing may be noticed in the soap, and to avoid any loss from this cause or any creeping of the soap up the sides of the flask, it should be removed from the bath and shaken to and fro until the frothing disappears. The last traces of alcohol vapor may be removed from the flask by waving it briskly, mouth down, to and fro.

Dissolving the soap.—After the removal of the alcohol the soap should be dissolved by adding 100 cc. of recently boiled distilled water, warming on the steam bath with occasional shaking, until solution of the soap is complete.

Setting free the fatty acids.—When the soap solution has cooled to about 60° or 70° the fatty acids are separated by adding 40 cc. of the dilute sulphuric acid solution mentioned above.

Melting the fatty acid emulsion.—The flask should now be re-stoppered as in the first instance, and the fatty acid emulsion melted by replacing the flask on the steam bath. According to the nature of the fat examined, the time required for the fusion of the fatty acid emulsions may vary from a few minutes to several hours.

The distillation.—After the fatty acids are completely melted, which can be determined by their forming a transparent oily layer on the surface of the water, the flask is cooled to room temperature, and a few pieces of pumice stone added. The pumice stone is prepared by throwing it, at a white heat, into distilled water, and keeping it under water until used. The flask is now connected with a glass condenser slowly heated with a naked flame until ebullition begins, and then the distillation continued by regulating the flame in such a way as to collect 110 cc. of the distillate in, as nearly as possible, thirty minutes. The distillate should be received in a flask accurately graduated at 110 cc.

Titration of the volatile acids.—The 110 cc. of distillate, after thorough mixing, are filtered through perfectly dry filter paper and collected in a flask graduated at 100 cc. The 100 cc. of the filtered distillate are poured into a beaker holding from 200 to 250 cc., 0.5 cc. phenolphthalein solution added, and decinormal barium hydrate run in until a red color is produced. The contents of the beaker are then returned to the measuring flask to remove any acid remaining therein, poured again into the beaker, and the titration continued until the red color produced remains apparently unchanged for two or three minutes. The number of cubic centimeters of decinormal $Ba(OH)_2$ required should be increased by one-tenth.

ALTERNATE METHOD OF DETERMINING VOLATILE ACIDS.

Saponification without the use of alcohol.—To avoid the danger of loss from the formation of ethers, and the trouble of removing the alcohol after saponification, the fat may be saponified with a solution of caustic potash in a closed flask without using alcohol. The operation is carried on exactly as indicated above for saponification in a closed flask, using caustic potash solution instead of soda, and omitting the operation for volatilizing the alcohol. The caustic potash is prepared as follows: Dissolve 100 grams of the purest potassium hydrate in 58 grams of hot distilled water. Allow to cool in a stoppered vessel, decant the clear solution, and preserve in a vessel out of contact with the air. For the saponification use 2 cc. of the caustic-potash solution, which are poured on the fat after it has solidified in the flask. Great care must be taken that none of the fat is allowed to rise on the sides of the saponifying flask to a point where it can not be reached by the alkali. During the process of saponification the flask can only be very gently rotated in order to avoid the difficulty mentioned. This process is not recommended in any except a closed flask with round bottom. In the subsequent solution of the soap use only 80 cc. of distilled water, and in setting free the fatty acids use 60 cc. of the dilute sulphuric acid. In other respects the distillation is conducted as previously described. Potash is used instead of soda, so as to form a softer soap, and thus allow a more perfect saponification.

The saponification may also be conducted as follows: The alkali and fat in the melted state are shaken vigorously in the saponification flask until a complete emulsion is secured. The rest of the operation is then conducted as above.

IODIN ABSORPTION NUMBER.

Reagents.

(1) Iodin solution.—Dissolve 25 grams of pure iodin in 500 cc. of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc. of 95 per cent alcohol. The last solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using.

(2) Decinormal sodium thiosulphate solution.—Take 24.6 grams of chemically pure sodium thiosulphate freshly pulverized as finely as possible and dried between filter or blotting paper. Make this up to 1,000 cc. at the temperature at which the titrations are to be made.

(3) Starch paste.—One gram of starch boiled in 200 cc. of distilled water for ten minutes and cooled to room temperature.

(4) Solution of potassium iodid.—One hundred and fifty grams of potassium iodid dissolved in water and made up to 1 liter.

(5) Solution of potassium bichromate.—Dissolve 3.874 grams of chemically pure potassium bichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made.

Manipulation.

Standardizing the sodium thiosulphate solution.—Run 20 cc. of the potassium bichromate solution, to which has been added 10 cc. of the solution of potassium iodid, into a glass-stoppered flask. Add to this 5 cc. of strong hydrochloric acid. Allow the solution of sodium thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste and with constant shaking continue to add the sodium thiosulphate solution until the blue color just disappears. The number of cubic centimeters of thiosulphate solution used multiplied by 5 is equivalent to 1 gram of iodin.

Example.—Twenty cc. $K_2Cr_2O_7$ solution required 16.2 cc. sodium thiosulphate; then $16.2 \times 5 = 81 =$ number cubic centimeters of thiosulphate solution equivalent to 1 gram of iodin. Then 1 cc. thiosulphate solution $= 0.0124$ gram of iodin. Theory for decinormal solution of sodium thiosulphate, 1 cc. $= 0.0127$ gram of iodin.

Weighing the sample.—About 1 gram of butter fat is to be weighed in a glass-stoppered flask holding about 300 cc., with the precautions mentioned for weighing the fat for determining volatile acids.

Absorption of iodin.—The fat in the flask is dissolved in 10 cc. of chloroform. After complete solution has taken place, 30 cc. of the iodin-mercuric chlorid solution is added. The flask is now placed in a dark place and allowed to stand, with occasional shaking, for three hours.

Titration of the unabsorbed iodin.—100 cc. of distilled water are added to the contents of the flask, together with 20 cc. of the potassium iodid solution. Any iodin which may be noticed upon the stopper of the flask should be washed back into the flask with the potassium iodid solution. The excess of iodin is now taken up with the sodium thiosulphate solution, which is run in gradually, with constant shaking, until the yellow color of the solution has almost disappeared. A few drops of starch paste are then added, and the titration continued until the blue color has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken, so that any iodin remaining in solution in the chloroform may be taken up by the potassium iodid solution in the water. A sufficient quantity of sodium thiosulphate solution should be added to prevent a reappearance of any blue color in the flask for five minutes.

Setting the value of the iodin solution by the thiosulphate solution.—At the time of adding the iodin solution to the fats, two blank flasks of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodin solution is used.

Example—Blank determinations.

(1) 30 cc. iodin solution required 46.4 cc. sodium thiosulphate solution.
(2) 30 cc. iodin solution required 46.8 cc. of sodium thiosulphate solution.
Mean, 46.6.

Per cent of iodin absorbed.

Weight of fat taken ....grams..	1.0479
Quantity of iodin solution used........................cubic centimeters..	0.30
Thiosulphate equivalent to iodin used .....:........................do....	46.6
Thiosulphate equivalent to remaining iodin...........................do....	14.7
Thiosulphate equivalent to iodin absorbed............................do....	31.9

Per cent of iodin absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$.

ELECTIVE METHODS OF BUTTER ANALYSIS.

The elective methods of butter analysis remain as described in Bulletin No. 31, pages 200–202.

METHODS FOR MILK ANALYSIS.

DETERMINATION OF WATER.

Evaporate 1 to 2 grams of milk in a tared flat dish containing from 15 to 20 grams of pure dry sand, or without sand, until apparently dry. Dry and heat for one hour at the temperature of boiling water. Cool in a desiccator, and weigh rapidly to avoid absorption of hygroscopic moisture.

DETERMINATION OF TOTAL NITROGENOUS MATTER.

Place in a Kjeldahl digestion flask a known weight (about 5 grams) of milk, and proceed, without evaporation, exactly as described for this method under nitrogen.

DETERMINATION OF TOTAL SOLIDS AND FAT—BABCOCK ASBESTOS METHOD.

Provide a hollow cylinder of perforated sheet metal, 60 mm. long and 20 mm. in diameter, closed 5 mm. from one end by a disc of the same material. The perforations should be about 0.7 mm. in diameter and about 0.7 mm. apart. Fill loosely with 1.5 to 2.5 grams of freshly ignited wooly asbestos, free from fine and brittle material, cool in a dessiccator and weigh. Introduce a weighed quantity of milk (3 to 5 grams) and dry at 100° C. to constant weight for the determination of total solids. Extract with anhydrous ether until fat is removed, evaporate the ether, dry the fat at 100° C. and weigh. The fat may also be determined by difference, drying the extracted cylinders at 100° C.

DETERMINATION OF FAT—PAPER-COIL METHOD.

Coils made of thick filter paper, cut into strips 6.25 by 62.5 mm. are thoroughly extracted with ether and alcohol, or the weight of the extract corrected by a constant obtained for the paper. If this latter method is used a small amount of anhydrous sodium carbonate should be added. From a weighing bottle about 5 grams of milk are transferred to the coil by a pipette, care being taken to keep the end of the coil held in the fingers dry. The coil, dry end down, on a piece of glass, is dried at the temperature of boiling water for one hour, or, better, dried in hydrogen at the temperature of boiling water, transferred to an extraction apparatus, and extracted with absolute ether or petroleum spirit boiling at about 45°. The extracted fat is dried in hydrogen and weighed.

If the milk is sour add about 10 per cent, by weight, of strong ammonia water, and a small quantity of anhydrous sodium carbonate, correcting the results for the ammonia added.

DETERMINATION OF SUGAR.

Reagents.

(1) *Basic lead acetate,* sp. gr. 1.97.—Boil a saturated solution of sugar of lead with an excess of litharge and make it of the strength indicated above. 1 cc. of this will precipitate the albumens in 50 to 60 cc. of milk.

(2) *Acid mercuric nitrate.* Dissolve mercury in double its weight of nitric acid, sp. gr. 1.42. Add to the solution an equal volume of water. 1 cc. of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodid with acetic acid.*—KI, 33.2 grams, $HgCl_2$, 13.5 grams, $C_2H_4O_2$, 20 cc. H_2O, 64 cc.

Apparatus.

(1) Pipettes marked at 59.5, 60, and 60.5 cc. (2) Sugar flasks marked at 102.4 cc. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.—(1) The room and milk should be kept at a constant temperature.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flasks

(3) If the specific gravity be 1.026, or nearly so, place 60.5 cc. in the sugar flask. Add 1 cc. of mercuric nitrate solution, or 30 cc. of mercuric iodid solution, and fill to the 102.4 cc. mark. The precipitated albumen occupies a volume of about 2.44 cc. Hence the milk solution is really 100 cc. If the specific gravity is 1.030, use 60 cc. of milk. If the specific gravity is 1.034, use 59.5 cc. of milk.

(4) Fill up to the mark in the 102.4 cc. flask, shake well, filter, and polarize.

NOTES. In the above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100 cc. solution to read 100 degrees on the cane sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar, whatever instrument is employed.

Since the quantity of milk taken is three times 20.56 grams, the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200-mm. tube is used.

If a 400-mm. tube is employed, divide the reading by 6; if a 500-mm. tube is used, divide by 7.5.

By using a flask graduated at 102.4 cc. for 60 cc. no correction for volume of precipitated casein need be made. In no case is it necessary to heat the sample before polarizing.

ALTERNATE METHOD FOR SUGAR.

The sugar may also be determined, either gravimetrically or volumetrically, by alkaline copper solution, as described under sugar analysis.

DETERMINATION OF ASH.

In a weighed dish put 20 cc. of milk from a weighing bottle; add 6 cc. of HNO_3, evaporate to dryness, and burn at low red heat until ash is free from carbon.

PROVISIONAL METHODS OF CHEESE ANALYSIS.

SAMPLING.

Where the cheese can be cut, a narrow wedge reaching from the edge to the center of the cheese will more nearly represent the average composition of the cheese than any other sample. This may be chopped quite fine, with care to avoid evaporation of water, and the several portions for analysis taken from the mixed mass. When the sample is taken with a cheese tryer, a plug taken perpendicular to the surface one-third of the distance from the edge to the center of the cheese should more nearly represent the average composition than any other. The plug should either reach entirely through or only half way through the cheese. For inspection purposes the rind may be rejected, but for investigations, where the absolute quantity of fat in the cheese is required, the rind should be included in the sample. It is well, when admissible, to take two or three plugs on different sides of the cheese and, after splitting them lengthwise with a sharp knife, take portions of each for the different determinations.

DETERMINATION OF WATER.

From 5 to 10 grams of cheese should be taken, and placed in thin slices in a weighed platinum or porcelain dish which contains a small quantity of freshly ignited asbestos, to absorb the fat which may run out of the cheese. This is then heated in a boiling water oven for ten hours and weighed; the loss in weight shall be considered as water. Or if preferred the dish may be placed in a desiccator over concen-

trated sulphuric acid and dried to constant weight. In some cases this may require as much as two months. The acid should be renewed when the cheese has become nearly dry.

DETERMINATION OF ASH.

The dry residue from the water determination may be taken for the ash. If the cheese be rich the asbestos will be saturated therewith. This may be ignited carefully and the fat allowed to burn off, the asbestos acting as a wick. No extra heat should be applied during this operation, as there is danger of spurting. When the flame has died out the burning may be completed in a muffle at low redness. When desired, the salt may be determined in the ash in the manner specified for butter.

DETERMINATION OF FAT.

Take 5 to 10 grams of cheese and grind it in a small mortar with about twice its weight of anhydrous copper sulphate. The grinding should continue until the cheese is finely pulverized and evenly distributed throughout the mass, which will have a uniform light blue color. This mixture is transferred to a glass tube which has a strong filter paper, supported by a piece of muslin, tied over one end. Put a little of the clean anhydrous copper sulphate into the tube next to the filter before introducing the mixture containing the cheese. On top of the mixture place a tuft of ignited asbestos, and place the tube in a continuous extraction apparatus and extract with anhydrous ether for fifteen hours. Dry the fat obtained at 100° to constant weight.

DETERMINATION OF CASEIN.

Make a determination of nitrogen by the Kjeldahl method, taking about 2 grams of cheese, and multiply the result by 6.25 for casein.

OTHER CONSTITUENTS.

The sum of the percentages of the different constituents, determined as above, subtracted from 100 will give the amount of organic acids, milk sugar, etc., in the cheese.

SOIL AND ASH ANALYSIS.—PROVISIONAL METHODS FOR THE YEARS 1893 AND 1894.

SOIL.

SAMPLING.

The soil selected should be, as far as possible, in its natural condition, not modified by recent applications of manure, or changed by the transporting action of water or wind. Surface accumulations of decaying leaves, etc., should be removed before taking the sample. To eliminate accidental variations in the soil, select specimens from five or six places in the field which seem to be fair averages of the soil, remove 2 or 3 pounds of the soil, taking it down to the depth of 6 to 9 inches, unless there is a change from top soil to subsoil at a less depth, so as to include the whole depth of the soil. Mix these soils intimately, remove any stones, shake out all roots and foreign matters, and dry the soil until it becomes friable. Break down any lumps in a mortar with wooden pestle, but avoid pulverizing any mineral fragments; pass 8 to 10 pounds of the soil through a 1 mm. sieve, rejecting all pebbles and material too coarse to pass through the sieve, noting the percentage amount of soil so rejected. If the amount equals or exceeds 20 per cent this should be pulverized to pass through a 2 mm. sieve, and in this state submitted to analysis. Once

more mix intimately the sifted soil. Expose in thin layers in a warm room till thoroughly air-dry (or dry it in air bath at temperature of 40°), place 6 to 8 pounds in a clean bottle, with label of locality and date, and cork tightly.

The soil is rapidly dried to arrest nitrification; it is not heated above 40° lest there be dissipation of ammonia compounds, or a change in the solubility of the soil. The normal limit to which the soil may be heated in place by the sun's rays should not be exceeded in preparing a soil for an agricultural chemical analysis.

HYGROSCOPIC MOISTURE.

Place 5 grams of air-dry soil in a flat-bottomed and tared platinum dish; heat in air bath to 110° for eight hours; cool in a desiccator, and weigh; repeat the heating, cooling, and weighing at intervals of an hour till constant weight is found, and estimate the hygroscopic moisture by the loss of weight. Weigh rapidly to avoid absorption of moisture from the air.

WATER-HOLDING POWER.

In the throat of a clean 3-inch glass funnel place a very small filter, just large enough to prevent the soil from running through the stem; wet the filter and add 100 grams of the air-dry soil; from a graduate containing 100 cc. pour water on the soil till it is thoroughly wet and a few drops pass through; let it stand undisturbed till no more water flows from the soil, the funnel being covered with a glass plate to prevent evaporation. Return the water which has filtered through to the graduate. The number of cc. taken up by the soil will show the percentage capacity of the soil to hold water.

In using this process certain precautions are necessary to secure uniform results. The soil should be simply poured into the funnel, and not pressed or compacted in any way. When water is poured upon the soil, no disturbing influence, no handling or shaking of the soil, should take place.

CAPILLARY POWER OF SOILS.

This is determined by filling a long glass tube with soil, placing the lower end in water, and marking the height to which the water ascends, as shown by the changed color of the soil in the tube.

VOLATILE MATTER.

The platinum crucible and 5 grams of soil used to determine the hygroscopic moisture may be used to determine the volatile matter. Heat the crucible and dry soil to low redness. The heating should be prolonged till all organic material is burned away, but below the temperature at which alkaline chlorids volatilize. Moisten the cold mass with a few drops of a saturated solution of ammonium carbonate, dry, and heat to 150° to expel excess of ammonia. The loss in weight of the dry soil represents organic matter, water of combination, salts of ammonia, etc.

WATER-SOLUBLE MATERIALS OF THE SOIL.

To prepare a water extract of the soil, a percolator of glass or tin may be employed. It should be large enough to hold one kilo of soil.

By means of a perforated rubber stopper connect the neck of the percolator with a two-necked bottle of 2 liters capacity, pour sufficient distilled water (ammonia free) on the soil to moisten it all, and let the whole stand undisturbed for half an hour, then add more pure distilled water, and if the filtration is too slow use the filter pump, till a liter of filtrate is secured. If the soil extract is cloudy, filter through a plain filter.

(1) *Soluble solids.*—Evaporate 100 cc. to dryness on the water bath in a tared

dish to determine the percentage of water-soluble materials in the soil; each gram of residue representing 1 per cent of such materials. Test this dry residue for nitrates by pouring over it 10 cc. of pure H_2SO_4, holding in solution 3 or 4 milligrams of brucin sulphate.

(2) *Chlorids.*—Titrate 100 cc. with standard decinormal silver nitrate with two drops of a solution of K_2CrO_4 as an indicator. Titrate in a white porcelain dish and view the reaction through a yellow glass plate of such tint as will eliminate the color of the chromic solution. The reaction will then be sharply defined.

(3) *Sulphates.*—Precipitate the sulphates in 100 cc. of the soil extract with $BaCl_2$, in presence of a few drops of HCl.

Reserve the rest of the water solution for the estimation of nitrates.

ACID-SOLUBLE MATERIALS.

In the following scheme for soil analysis it is recommended to use the air-dry soil from the sample bottle for each separate investigation. A determination made once for all of hygroscopic moisture and of water of combination on a separate specimen of air-dry soil will afford corrections for all the other samples used. It is not desirable to ignite the soil before analysis or to heat it so as to change its chemical properties.

(1) *Acid digestion of the soil.*—Place 10 grams of the air-dry soil in a 150 to 200 cc. Bohemian glass flask, add 100 cc. of pure HCl of sp. gr. 1.115, insert the stopper, wire it securely, place in a steam bath, and digest for thirty-six hours at the temperature of boiling water. Pour the contents of the flask into a small beaker, wash out with distilled water, add the washings to the contents of the beaker, and filter through a washed filter. Residue is the amount insoluble in hydrochloric acid. Add a few drops of HNO_3 to the filtrate, and evaporate to dryness on the water bath; take up with hot water and a few drops of HCl, and again evaporate to complete dryness. Take up as before, and filter into a liter flask, washing with hot water. Cool and make up to mark. This is solution "A." The residue is soluble silica.

(2) *Ferric oxid, alumina, and phosphoric acid.*—To 100 cc. or 200 cc., according to the probable amount of iron present, of the solution A, add NH_4OH to alkaline reaction (avoiding excess), to precipitate ferric and aluminic oxids and phosphates. Expel the excess of ammonia by boiling, allow to settle, decant the clear solution through a filter; add to the flask 50 cc. of hot distilled water, boil, settle, and decant as before. After pouring off all the clear solution possible dissolve the residue with a few drops of HCl with heat, and add just enough NH_4HO to precipitate the oxids. Wash by decantation with 50 cc. of distilled water, and then transfer all the precipitate to the filter and wash with hot distilled water till the filtrate becomes free from chlorids. Save the filtrate and washings which form solution B. Dry the filter and precipitate at 110°, transfer the precipitate to a tared platinum crucible, burn the filter and add the ash to the precipitate, heat the whole red hot, cool in desiccator, and weigh. The increase of weight, minus the ash of filter and the phosphoric acid (found in a separate process), represents the weight of the ferric and aluminium oxids.

(3) *Ferric oxid.*—Precipitate 100 cc. of solution A, as under 2, except that only one precipitation is made; wash with hot water; dissolve while wet in dilute H_2SO_4; reduce with zinc and estimate ferric oxid by a standard solution of potassium permanganate. To prepare the potassium permanganate solution, dissolve 3.156 grams of pure crystallized potassium permanganate in 1,000 cc. of distilled water, and preserve in a ground-glass-stoppered bottle, shielded from the light. Standardize this solution with pure ferrous sulphate or ammonio-ferrous sulphate or oxalic acid.

The weight of ferric oxid deducted from ferric oxid and alumina (12), with corrections for filter ash and phosphoric acid, will give the weight of alumina in 2 grams of air-dry soil.

(*4*) *Phosphoric acid.*—This may be estimated in the above iron solution if the soil is sufficiently rich, by the molybdate method, given under fertilizers; or if the quantity of soil represented in the iron solution is not sufficient, a fresh portion of solution A may be taken, and the phosphoric acid determined directly by the molybdate method.

(*5*) *Manganese.*—Concentrate the filtrate and washings (solution B) to 200 cc. or less; add NH_4OH to alkalinity; add bromin water and heat to boiling, keeping beaker covered with watch crystal; as the bromin escapes the beaker is allowed to cool somewhat, ammonia and bromin water again added, and heated as before.

This process is continued until the manganese is completely precipitated, which requires from thirty to sixty minutes, and the solution filtered while still warm, the precipitate washed, dried, ignited, and weighed; estimate as Mn_3O_4.

(*6*) *Lime.*—If no manganese is precipitated, add to solution B, or the filtrate and washings from (5) 20 cc. of a strong solution of NH_4Cl and 40 cc. of saturated solution of $(NH_4)_2C_2O_4$ to completely precipitate all the lime as oxalate and convert the magnesia into soluble magnesium oxalate. Heat to boiling and let stand for six hours till the calcium oxalate settles clear, decant the clear solution on a filter, pour 50 cc. of hot distilled water on the precipitate and again decant the clear solution on the filter, transfer the precipitate to the filter, and wash it free from all traces of oxalates and chlorids. Dry, and ignite the precipitate over the blast lamp until it ceases to lose weight, weigh and estimate as CaO; carefully moisten with H_2SO_4, heat the inclined covered crucible gently to avoid loss, then intensely, and weigh as $CaSO_4$

(*7*) *Magnesia.*—Concentrate the filtrate and washings (from 6) to 200 cc., place in a half-liter Erlenmeyer flask, add 30 cc. of a saturated solution of Na_2HPO_4 and 20 cc. of concentrated NH_4HO, cork the flask, and shake violently at intervals of a few minutes till crystals form, then set the flask in a cool place for twelve hours. Filter off the clear liquid through a tared Gooch filter, transfer the precipitate to the filter, and wash with dilute ammonium hydrate (1 : 3) till the filtrate is free from phosphates; dry and ignite the crucible, at first gently and then intensely, to form magnesium pyrophosphate. The increase of weight $\times$ 0.36024 $=$ MgO. By using an Erlenmeyer flask free from scratches and marks, and shaking violently instead of stirring with a glass rod, the danger is almost entirely avoided of crystals adhering to the sides of the vessel; but if crystals do adhere they are readily removed by a rubber-tipped glass rod.

(*8*) *Sulphuric acid.*—Evaporate 200 cc. of solution A (1) nearly to dryness on a water bath to expel the excess of acid; then add 100 cc. of distilled water; heat to boiling and add 10 cc. of a solution of $BaCl_2$, and continue the boiling for five minutes. When the precipitate has settled pour the clear liquid on a tared Gooch filter, treat the precipitate with 50 cc. of boiling water, and transfer the precipitate to the filter and wash with boiling water till the filtrate is free from chlorids. Dry the filter and ignite strongly. The increase in weight is barium sulphate, which multiplied by 0.34331 $=$ SO_3 in 2 grams of air-dry soil.

(*9*) *Potash and soda.*—[To another portion of 200 cc. of solution add $BaCl_2$ in slight excess, and make alkaline with ammonia to precipitate sulphuric and phosphoric acids, ferric oxid, etc. Then precipitate the calcium and barium by ammonium oxalate.[1]] Evaporate the filtrate and washings to dryness, heat to a low red heat to decompose oxalates and expel ammonia salts, dissolve in 25 cc. of distilled water, filter and wash the precipitate, add to the filtrate and washings 10 cc. of baryta water, and digest for an hour. Filter and wash the precipitate, add ammonium carbonate to the filtrate to complete precipitation of baryta, filter and wash this precipitate. Evaporate the filtrate and washings in a tared platinum dish, gently ignite the residue to expel ammonia salts, cool and weigh. The increase of weight represents the potassium and sodium chlorids in 2 grams of air-dry soil.

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[1] NOTE BY THE SECRETARY.—The sentences between brackets were inserted to repair an evident omission.

Separate and estimate the potassium chlorid by platinic chlorid according to the method given for potash determination.

Subtract the weight of potassium chlorid as thus found from the weight of potassium chlorid and sodium chlorid. The difference represents sodium chlorid.

Alternate method.—For an alternate method for alkalis, use J. Lawrence Smith's method as given in Crookes' Select Methods, second edition, pp. 28 to 40.

(10) *Other alkali metals.*—The salts of lithium, cesium, and rubidium are occasionally found in very small amounts in soils. The agricultural uses of these salts are still in question, and their amount is too small to admit of quantitative estimation. A quantitative examination may be made by the spectroscope with the water-soluble materials evaporated to dryness and dissolved with 2 or 3 drops of HCl. Test by the spectroscope with a platinum wire in a Bunsen flame.

<center>NITROGEN OF THE SOIL.</center>

The nitrogen compounds in the soil are usually placed in three classes:

1. The nitrogen combined with oxygen as nitrates or nitrites, existing as soluble salts in the soil.

2. The nitrogen combined with hydrogen as ammonia, or organic nitrogen easily convertible into ammonia. The ammonia may exist as salts, or be occluded by hydrated ferric or aluminum oxids and organic matter in the soil.

3. The inert nitrogen of the soil or the humus nitrogen.

Active soil nitrogen.—The material proposed for reducing the nitrates to ammonia, and at the same time to bring ammonia salts and organic nitrogen into a condition for separation by distillation, is sodium amalgam. Liquid sodium amalgam may be readily prepared by placing 100 cc. of mercury in a flask of half a liter capacity, covering the warmed mercury with melted paraffin and dropping into the flask at short intervals pieces of metallic sodium the size of a large pea (taking care that the violence of the reaction does not project the contents from the flask), till 6.75 grams of sodium have combined with the mercury. The amalgam contains 0.5 per cent of sodium and may be preserved indefinitely under the covering of paraffin.

To estimate the active soil nitrogen, weigh 50 grams of air-dry soil and place it in a clean mortar. Take 200 cc. of ammonia-free distilled water, rub up the soil with a part of the water to a smooth paste, transfer this to a flask of 1 liter capacity, washing the last traces of the soil into the flask with the rest of the water. Add 25 cc. of the liquid sodium amalgam and shake the flask so as to break the sodium amalgam into small globules distributed through the soil. Insert a stopper with a valve, and set aside in a cool place for twenty-four hours. Pour into the flask 50 cc. of milk of lime and distill, on a sand bath, 100 cc. into a flask containing 20 cc. of decinormal sulphuric acid, and titrate with decinormal soda solution, using dimethyl-orange as indicator. Estimate the nitrogen of the ammonia found as active soil nitrogen.

If the ammonia produced is too small in amount to be readily estimated volumetrically, determine the ammonia by Nesslerizing the distillate.

Estimation of nitrates in the soil.—When it is desired to estimate separately the nitrates in the soil the following method may be used: Evaporate 100 cc. of the soil extract to dryness on the water bath; dissolve the soluble portion of the residue in 100 cc. of ammonia-free distilled water, filtering out any insoluble residue, place the solution in a flask, and add 10 cc. of liquid sodium amalgam, insert stopper with valve, set it aside to digest in a cool place for twenty-four hours, add 50 cc. of milk of lime, distil and titrate as in 22, and estimate the nitrogen as N_2O_5.

Nesslerizing may be substituted for titration when the amount of nitrates is small.

An approximate estimation of the amount of nitrates will be of value in determining which method of estimation to use. This may be done by evaporating a measured quantity of the soil extract say 5 cc., on a porcelain crucible cover on a steam bath or radiator, having first dissolved a minute fragment of pure brucin

sulphate in the soil extract. When dry pour over the residue concentrated sulphuric acid, free from nitrates, and observe the color reactions produced.

If the nitrate (reckoned as KNO_3) left upon evaporating the quantity of water taken does not exceed the two-thousandth part of a milligram, only a pink color will be developed by adding the sulphuric acid; with the three-thousandth part of a milligram, a pink with faint reddish lines; with the four-thousandth part, a reddish color; with the five-thousandth part, a red color.

By increasing or diminishing the amount of soil extract evaporated to secure a color reaction of a certain intensity, an approximate estimate may be made of the amount of nitrates present.

Blank experiments to test the acid and the brucin sulphate will be required before confidence can be placed in such estimations.

Total nitrogen of soils.—The total nitrogen of soils may be determined by the usual combustion with soda-lime, but this process is often unsatisfactory because of the large amount of material required when the organic matter or humus is small in amount.

A modification of the Kjeldahl method is more easy to carry out and gives results equally satisfactory. Place 20 grams of soil in a Kjeldahl flask, and add 20 cc. of sulphuric acid (free from ammonia) holding in solution 1 gram of salicylic acid. (If the soil contains much lime or magnesia in the form of carbonate, enough more sulphuric acid must be added to secure a strongly acid condition of the contents of the flask. Add gradually 2 grams of zinc dust, shaking the contents of the flask to secure intimate mixture. Place the flask in a sand bath and heat till the acid boils, and maintain the boiling for 10 minutes. Add 1 gram of mercury and continue the boiling for 1 hour, adding 10 cc. of sulphuric acid if the contents of the flask are likely to become solid. Cool the flask and wash out the soluble materials with 200 cc. of pure water, leaving the heavy earthy materials. Rinse the residue with 100 cc. of water, and add this to the first washings. Place this soluble acid extract in a liter digestion flask, add 35 cc. of a solution of potassium sulphid, and shake the flask to secure intimate mixture of the contents. Introduce a few fragments of granulated zinc, pour in 75 cc. of a saturated solution of caustic soda, connect the flask with a condenser, and distill 150 cc. into a flask containing 20 cc. of acid, using the same acid and alkali for titration used in Kjeldahl method under fertilizers.

Enter the nitrogen found in this operation as total soil nitrogen.

The difference between the total soil nitrogen and the active soil nitrogen will express the inert nitrogen of the soil.

CARBON DIOXID.

This is determined as under ash analysis—using 1 to 5 grams—according to the amount of CO_2 present.

ASH.

PREPARATION OF ASH.

Before combustion the material must be thoroughly cleaned from all foreign matter, especially from adhering soil, wood, bark, roots, etc.

The combustion should be carried on at a comparatively low temperature, never reaching a full red heat, because of the danger of volatilizing alkaline chlorids, etc., nor in a strong draft of air, lest the lighter parts of the ash be carried away.

Combustion is best carried on in a flat platinum dish in a muffle.

With substances rich in silica and alkalis it is better to first char the substance. Wash with distilled water to remove soluble salts, then dry and incinerate the residue. Evaporate the watery extract and add this to the rest of the ash.

With substances rich in phosphates, *e. g.*, seeds and animal substances, char the material and remove salts by acetic acid, decant the acetic solution, wash with distilled

water, and then complete the combustion. Add the acetic solution and washings to the final ash, evaporate to dryness, and gently ignite the whole to decompose the acetates. In whatever way obtained the whole of the ash should be pulverized and intimately mixed before analysis.

ANALYSIS OF WOOD ASHES.

Pass 100 grams of dry ashes through a wire sieve (of 1 mm. mesh) to separate materials manifestly foreign, e. g., nails, broken glass and pottery, pebbles, etc., and estimate the per cent of such accidental materials. Pulverize any charcoal and semi-fused portions of ash remaining on the sieve, sift them and mix intimately with the sifted ashes, and preserve in stoppered bottles for analysis.

(1) *Moisture.*—Take 5 grams of the prepared sample in a tared platinum dish and heat to 110° in an air bath to constant weight.

(2) *Carbon.*—Heat this dried ash in a platinum dish till the ash is uniformly grayish white and there is no further loss of weight.

(3) *Sand and silica.*—Place 10 grams of ignited ash in a 150 cc. Bohemian flask, glass stoppered, measure out 100 cc. of HCl (specific gravity 1.115) and pour on the ash cautiously to prevent loss, and when all effervescence has ceased, add the rest of the acid; insert the glass stopper, wire it securely, and place in a steam bath for two hours; empty the flask into a platinum dish, wash with distilled water, adding the washings to the ash solution, and evaporate the whole to dryness on a water bath. Add 10 cc. of dilute HCl and 50 cc. of distilled water to the contents of the platinum dish, transfer the contents to a filter, wash with distilled water till the last drops of the filtrate are free from chlorids, dry and ignite the precipitate, and filter. If there are no grains of sand (revealed by grittiness when stirred with a glass rod), subtract the ash of the filter from the weight of this residue and estimate the rest as silica. If sand is present, boil the ignited and weighed residue in a strong solution of Na_2CO_3 to dissolve silica, wash by decantation to remove all sodium salts, dry, and weigh the sand. The difference between the weight of sand, and silica + sand, will give the weight of silica.

The filtrate and washings from sand and silica are made up to 1,000 cc. (solution C) and kept for analysis.

(4) *Phosphoric acid.*—Take 100 or 200 cc. of solution C and estimate P_2O_5 by the official method.

(5) *Carbonic acid.*—Heat 4 or 5 grams of ash in a muffle till all charcoal is consumed; cool in a desiccator, transfer 2 grams to a carbonic-acid apparatus. Estimate the CO_2 in any convenient form of apparatus.

Alternate method.—By Liebig's potash bulbs. The usual process of absorption by solution of KHO, weighing, etc.

(6) *Chlorin.*—Pour out the nitric-acid solution from the CO apparatus upon a filter, wash out the last traces of the solution, pass the soluble matters through the filter and wash the insoluble residue with water acidulated with HNO_3. To this filtrate add solution of $AgNO_3$ to complete precipitation of the chlorids, boil and set aside in a dark place until the precipitate has settled completely, filter through a Gooch crucible, and wash with hot water. Dry at 110°, and weigh the silver chlorid.

(7) *Alternate method for chlorin.*—Boil 10 grams of ash in 400 cc. of pure water for half an hour, transfer all to a measuring flask of 500 cc. capacity, wash the beaker, and add the washings to the flask, cool, make up the volume to 500 cc. and mix intimately; filter off through a dry filter 100 cc., add a drop of solution of phenolphthalein, and neutralize with dilute HNO_3 till only a faint pink color remains, add 2 drops of a strong solution of K_2CrO_4 and titrate with standard decinormal solution of $AgNO_3$ (16.956 grams $AgNO_3$ to 1,000 cc.). Every cubic centimeter of the standard silver solution equals 0.003546 gram of chlorin.

The reliability of this method will depend upon the accuracy with which neutralization by nitric acid has been made. The least trace of free acid or alkaline carbonate will vitiate the results.

(*8*) *Sulphuric acid.*—Take an aliquot part of solution A and determine as under soils.

(*9*) *Oxid of iron.*—Take 200 cc. of the original acid solution, nearly neutralize with ammonia, then add 1 gram of sodium acetate, and acetic acid in slight excess, boil to precipitate ferric phosphate, filter while hot, and wash the precipitate with boiling distilled water till the filtrate is free from chlorids. Dissolve the precipitate on the filter with dilute H_2SO_4 into a small Erlenmeyer flask, wash the filter, dry and ignite and add the ashes to the acid solution in the flask, reduce the ferric to ferrous salt by zinc or by a coil of magnesium wire till a drop of the solution gives no color with NH_4CNS. Pour off the solution of ferrous salt into a beaker, rinse the flask and add the rinsings to the beaker, add freshly-boiled distilled water to make 200 cc. of the solution, add 2 cc. of sulphuric acid, heat to 70°, and titrate with standard solution of permanganate, and estimate the iron as ferric oxid.

(*10*) *Manganese.*—If a qualitative test shows the presence of manganese, evaporate the filtrate and washings from iron determination to 200 cc. or less, and estimate as under soils.

(*11*) *Lime.*—Evaporate the filtrate and washings from ferric phosphate to 100 cc. To the hot solution add 20 cc. of concentrated solution of ammonium chlorid and 40 cc. of saturated solution of ammonium oxalate; boil the whole for ten minutes and then let it stand in a warm place for six hours; decant the clear liquid upon a filter, wash the precipitate twice by decantation, then bring the precipitate upon the filter, and wash it free from chlorids and oxalates, testing the washings by silver nitrate. Ignite and estimate as under soils.

(*12*) *Magnesia.*—Evaporate the filtrate and washings from the calcium oxalate to 200 cc., pour into a clean and unscratched Erlenmeyer flask of 500 cc. capacity, add 30 cc. of strong solution of $(NH_4)_2HPO_4$ and 50 cc. of strong ammonium hydrate, cork the flask and shake violently at intervals of a few minutes till crystallization is established, and then set aside for twelve hours in a cold place. If crystals should form on the sides of the flask they are readily detached by a rubber-tipped rod, Filter through a tared Gooch filter, wash the precipitate with ammonium hydrate, diluted with distilled water (1 to 3), till the filtrate is free from phosphates. Dry the precipitate, ignite, at first very gently and then intensely, with blast lamp, to convert $Mg_2NH_4PO_4$ into $Mg_2P_2O_7$. Cool in desiccator and weigh. The increase of weight × .36024 = Mg O, in 2 grams of air-dry soil.

(*13*) *Estimation of alkalis.*—Concentrate the filtrate and washings from (8) to 100 cc, add NH_4HO and $(NH_4)_2C_2O_4$ to complete precipitation of barium and calcium, filter, wash the precipitate, evaporate the filtrate and washings to dryness in a platinum dish, and ignite gently. Add to the residue concentrated solution of oxalate of ammonia, evaporate to dryness, and ignite gently. Dissolve residue in distilled water, filter from insoluble MgO, acidify the filtrate with HCl, and evaporate to dryness in a tared platinum dish and ignite gently. The increase of weight represents the potassium and sodium chlorids. Separate and estimate potassium by $PtCl_4$ in the usual way, and determine the sodium by difference.

The ash of mineral coal contains only a small amount of alkalis and phosphates, but a large amount of insoluble material, clay, etc. The value depends mostly upon the sulphate of lime and phosphate present. They are often decomposed with difficulty. It should be ground to fine powder, and 5 grams placed in the digestion flask with 50 cc. of HCl (sp. gr. 1.115), and digested in the steam bath for six hours, and the soluble portion analyzed in the usual way.

METHODS OF ANALYSIS OF FERMENTED LIQUORS.

I. *Specific gravity.*—This determination is made with a pycnometer, or a Westphal balance controlled by a pycnometer, at a temperature of 15.5°.

II. *Alcohol.*—Weigh out about 100 grams of wine and distill in a proper distilling apparatus (provided with a glass condenser and mercury valve), after the addition

of 50 cubic centimeters of water. When about 100 cc. of distillate have come over the operation is stopped and the distillate weighed. Its specific gravity at 15.5° is then ascertained, by means either of a picnometer or a Westphal balance controlled by a pycnometer, and the corresponding per cent of alcohol by weight obtained from the official tables. This figure, multiplied by the weight of distillate, and the result divided by the weight of sample taken gives the per cent of alcohol by weight contained in the sample.

III. *Extract.*—Fifty cc. (weighed), or in case of sweet wines a smaller amount, is evaporated on a water bath to a sirupy consistency in a platinum dish 85 millimeters in diameter and of about 75 cc. The residue is heated for two and one-half hours in a drying oven at 100°. In the case of sweet wines, that is, those containing more than 4 per cent of solids, not more than 10 cc. (weighed) should be taken.

Alternate method for extract in sweet wines.—Measure out carefully a definite volume of wine, transfer to an evaporating dish, and evaporate on a water bath until its volume is reduced to about one-third. Transfer the dealcoholized liquid to the graduated vessel in which it was originally measured out, rinse the dish carefully and make it up to the same volume with water, mix the liquid thoroughly, and ascertain its specific gravity, either by a pycnometer or by an accurately standardized Balling or Brix spindle. The per cent of total solids, in case the picnometer is used, may be taken from the official tables herewith.

IV. *Acidity* (total acid constituents of the wine expressed as tartaric acid).—If carbonic acid is present, expel by shaking. Titrate with dilute alkali solution. The neutral point is determined by adding a drop of the solution to delicate litmus paper.

V. *Volatile acids* (expressed as acetic acid).—Distill in a current of steam, and titrate the carefully condensed distillate with standard alkali (decinormal).

VI. *Glycerol.*—(1) This is determined in dry wines as follows: The alcohol is driven off from 100 cc. of wine, lime or magnesia is added, and then evaporated to dryness. The residue is boiled with 90 per cent alcohol, filtered, and the filtrate evaporated to dryness. This residue is dissolved in 10 to 20 cc. of alcohol, 15 to 30 cc. of ether is added, and the mixture allowed to stand until it is clear. It is then decanted from the sticky precipitate into a glass-stoppered weighing bottle, evaporated to constant loss of weight, and weighed.

(2) The following method is employed for sweet wines: 100 cc. of wine is measured into a porcelain dish and evaporated on the water bath to a sirupy consistency, mixed with 100 to 150 cc. of absolute alcohol, poured into a flask, ether added in the proportion of one and one-half volumes to each volume of alcohol used, the flask well shaken, and allowed to stand until the liquid becomes clear. This is then poured off, and the residue again treated with a mixture of alcohol and ether. The liquids are mixed, the alcohol and ether driven off, the residue dissolved in water, and treated as in (1).

(3) It is necessary to test the glycerol from sweet wines for sugar, and if any is present it must be estimated by Soxhlet's method, and its weight subtracted from that of the glycerol.

VII. *Sugar.*—This is to be determined by Soxhlet's method. The presence of unfermented cane sugar is to be shown by inversion.

The wine should not contain more than 1 per cent of sugar.

Preliminary test.—100 cc. of wine accurately neutralized with normal soda solution is dealcoholized by evaporation to two-thirds, and the original volume restored with water (red wines are decolorized with pure animal charcoal); 25 cc. of copper solution and 25 cc. of Seignette salt solution are heated to boiling in a deep porcelain evaporating dish; wine is added from a burette till the fluid, after two minutes' boiling, no longer has a blue color. This gives an approximate estimate of the sugar present, and from this we can dilute the wine to very nearly 1 per cent of sugar.

Final determination.—25 cc. of wine, as in preliminary test, 25 cc. of Seignette solution, and 23 to 24 cc. of diluted wine (approximately 1 per cent), are heated two

minutes to boiling, the precipitated copper allowed to settle, and filtered through a double filter. If the filtrate is greenish, it contains copper; if the filtrate is yellow, it is acidulated, and after cooling a little acetic acid is added and two or three drops potassium ferrocyanid. If a reddish color is formed, copper is still present, and a sufficient amount of sugar has not been added to reduce the 50 cc. of Fehling's solution. The experiment is repeated, adding 1 to 2 cc. more wine if copper was present in the filtrate; if no copper was in the filtrate, 1 cc. less of wine. The experiment is continued till filtrates are obtained containing a difference of 0.1 cc. of wine, one of which contains copper solution and one not. The mean of the two contains the sugar equivalent to 50 cc. of Fehling's solution; 50 cc. of Fehling's solution corresponds to 0.2375 grams of grape sugar.

Polarization.—(1) The wine is decolorized with lead subacetate.

(2) A slight excess of sodium carbonate is added to the filtrate from (1); 2 cc. of a solution of lead subacetate are added to 40 cc. of white wine, and 5 cc. to 40 cc. of red wine, the solution is filtered, and 1 cc. of a saturated solution of sodium carbonate added to 21.0 or 22.5 cc. of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and the results estimated in equivalents of Wild's polaristrobometer, with 200-millimeter tubes, after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotary power of less than 0.3° to the right shows that impure glucose has not been added.

(6) Wines rotating between 0.3° and 0.5° to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented, and their optical properties then examined.

VIII. *Tannin.*—Determine by the Neubauer and Löwenthal method.

100 cc. of wine is dealcoholized by continued boiling, and the lost weight restored by water. (1) 10 cc. of dealcoholized wine is transferred to a large porcelain evaporating dish, 1 liter of water added, also 10 cc. of dilute H_2SO_4, and then 20 cc. of indigo solution; titrate with $K_2Mn_2O_8$ solution, adding this reagent with great care (drop by drop) towards the end of the reaction. (2) 10 cc. of dealcoholized wine treated with bone black to remove tannin and coloring matter, after standing for a time, is filtered, washed with water, and diluted to 1 liter, and titrated with $K_2Mn_2O_8$ solution, as in (1) (viz, after adding 10 cc. of dilute H_2SO_4 and 20 cc. of indigo).

From the number of cubic centimeters of $K_2Mn_2O_8$ used in (2) subtract the number of cubic centimeters of $K_2Mn_2O_8$ required to oxidize the indigo, and the remainder will give the number of cubic centimeters of $K_2Mn_2O_8$ necessary to oxidize the matters other than tannin and color.

From the number of cubic centimeters of $K_2Mn_2O_8$ used in (1) take the value of the indigo used, and the remainder will be the number of cubic centimeters necessary to oxidize tannin, color, and other oxidizable matter. Subtract from this the number of cubic centimeters of $K_2Mn_2O_8$ from (2) after taking out the value of indigo, and the remainder will be the amount necessary to oxidize the tannin and color. (The amount required for color can be neglected.)

IX. *Potassium bitartrate.*—The determination of potassium bitartrate as such is to be omitted.

X. *Tartaric, malic, and succinic acids.*—(1) According to Schmidt and Hiepe's method.

To 200 cc. of wine evaporated one-half and allowed to cool, basic lead acetate is added to alkaline reaction. The precipitate is filtered off and washed with cold water till the filtrate shows only a slight lead reaction. The precipitate is washed from the filter with a wash bottle into a beaker and treated hot with H_2S till the lead is completely converted into sulphid, filtered hot, and washed with hot water. The filtrate and wash water are evaporated to 50 cc., neutralized with KHO, and again concentrated. An excess of $Ca(C_2H_3O_2)_2$ is added and allowed to stand four to six hours, with frequent stirring. It is then filtered and washed with water till the filtrate and

wash water amount to 100 cc. The calcium tartrate is then converted into caustic lime by strong ignition, and, according to quantity, 10 to 15 cc. of normal HCl added, and the solution diluted and retitrated very accurately. For every cubic centimeter of normal HCl saturated by the caustic lime, 0.075 grams of tartaric acid is to be set down, and to this 0.0286 grams added, representing the total tartaric acid present in 200 cc. wine.

The filtrate from the calcium tartrate is now evaporated to about 20 to 30 cc., allowed to cool, and three times the volume of 96 per cent alcohol added. After standing several hours the precipitate is collected upon a weighed filter, dried at 100° C., and weighed. This represents the lime salts of the malic and succinic acids, and the tartaric acid still remaining in solution, and of the sulphuric acid in the wine. This precipitate is now dissolved in a minimum of HCl, washed with hot water, and then precipitated hot with K_2CO_3, and the $CaCO_3$ filtered off. The filtrate now contains the potash salts of the above-mentioned acids. This is neutralized with acetic acid and evaporated to a small volume, and precipitated hot with $BaCl_2$. This precipitate of succinate and sulphate of barium is treated on the filter with dilute HCl; the filtrate now contains only the succinate of barium, is treated with H_2SO_4 and the Ba precipitated. (The $BaSO_4$ remaining on the filter is ignited and weighed.) The $BaSO_4$ obtained from the succinate solution is filtered off, washed, dried, ignited, and weighed; 223 parts of $BaSO_4$ correspond to 118 parts succinic acid. The succinic and sulphuric acids, as well as the tartaric acid remaining in solution, which was equal to 0.0286 grams, are to be calculated as lime salts and subtracted from the total weight of the lime precipitate. The remainder is calcium malate, of which 172 parts correspond to 134 parts of malic acid.

(2) Determination of tartaric acid according to the modified Berthelot-Fleury method.

Fifty cc. is measured and 10 cc. of same neutralized with caustic potash. The two liquids are now mixed, and one-fifth of the mixture, 10 cc., is taken, and 50 cc. of a mixture of alcohol and ether added and allowed to stand twenty-four hours, and the separated cream of tartar filtered off, dissolved in water, and titrated. The excess of cream of tartar above that obtained in the estimation of tartar alone corresponds to the free tartaric acid.

XI. *Coloring matter.*—(1) Only anilin dyes are to be looked for.

(2) Special attention is to be paid to the spectroscopic behavior of rosanilin dyes as obtained by shaking wines with amylic alcohol before and after saturation with ammonia.

XII. *Inorganic matter* (ash).—Burn in the ordinary manner in a flat platinum dish at as low a heat as possible; repeated moistening, drying, and heating to redness are advisable to get rid of all organic substances.

XIII. *Citric acid.*—Presence to be shown by a qualitative test, as barium citrate.

XIV. *Sulphuric acid.*—To be determined in the wine after adding hydrochloric acid.

XV. *Chlorin.*—To be determined in the nitric acid solution of the burnt residue by Volhard's method.

XVI. *Lime, magnesia, and phosphoric acid.*—These are determined in the ash fused with sodium hydrate and potassium nitrate, the phosphoric acid by the molybdenum method.

XVII. *Potash.*—Either in the wine ash, as the platinum double salt, or in the wine itself, by Kayser's method.

XVIII. *Gums.*—Presence shown by precipitation with alcohol; 4 cc. of wine and 10 cc. of 96 per cent alcohol are mixed. If gum arabic has been added, a lumpy, thick, stringy precipitate is produced; whereas pure wine becomes at first opalescent and then flocculent.

XIX. *Sulphurous acid.*—100 cc. of wine is distilled in a current of carbonic acid after the addition of phosphoric acid. The distillate carefully condensed is oxidized with bromin and the amount of H_2SO_4 determined.

TABLE I.—Correction to be applied to the reading of Balling's saccharometer for different temperatures.

Degree Balling.

Temp. °F.	1°	2°	3°	4°	5°	6°	7°	8°	9°	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°
50	.17	.17	.18	.19	.20	.20	.21	.21	.22	.22	.22	.23	.23	.24	.24	.24	.25	.25	.25	.26	.23
51	.16	.16	.17	.18	.19	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.25
52	.15	.15	.16	.17	.18	.16	.18	.19	.19	.19	.19	.19	.20	.20	.21	.21	.22	.22	.22	.22	.23
53	.14	.14	.14	.15	.16	.16	.16	.17	.17	.18	.18	.18	.18	.19	.19	.19	.19	.20	.20	.20	.20
54	.12	.12	.13	.14	.15	.15	.15	.16	.16	.16	.16	.17	.17	.17	.17	.17	.18	.18	.18	.18	.18
55	.07	.09	.09	.10	.11	.11	.11	.12	.12	.12	.12	.13	.13	.13	.14	.14	.14	.14	.14	.14	.15
56	.07	.07	.07	.08	.09	.09	.09	.09	.10	.10	.10	.10	.11	.11	.11	.11	.11	.11	.11	.11	.11
57	.06	.06	.06	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.09	.09	.09	.09	.10	.10	.10	.10
58	.05	.04	.04	.05	.05	.05	.05	.05	.06	.06	.06	.06	.06	.07	.07	.07	.07	.07	.07	.07	.07
59	.02	.02	.02	.02	.02	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
60																					
61	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
62	.06	.06	.07	.07	.08	.08	.08	.08	.08	.08	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09
63	.09	.10	.10	.11	.11	.11	.11	.11	.11	.11	.12	.12	.12	.12	.12	.12	.12	.12	.13	.12	.12
64	.12	.12	.13	.13	.14	.14	.14	.14	.15	.15	.15	.15	.19	.16	.17	.17	.17	.17	.17	.17	.17
65	.14	.15	.15	.16	.17	.17	.17	.17	.18	.18	.18	.19	.19	.19	.20	.20	.20	.20	.20	.20	.20
66	.17	.17	.18	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.24	.24	.24
67	.20	.21	.21	.22	.23	.23	.23	.24	.24	.25	.25	.25	.26	.26	.27	.27	.27	.27	.27	.27	.27
68	.22	.23	.24	.25	.26	.26	.27	.28	.28	.29	.29	.30	.30	.31	.31	.31	.31	.31	.31	.31	.31
69	.25	.26	.27	.28	.29	.29	.30	.31	.31	.32	.32	.33	.33	.34	.34	.34	.34	.33	.34	.34	.34
70	.29	.29	.30	.31	.32	.32	.33	.34	.34	.35	.35	.36	.36	.37	.37	.37	.37	.38	.38	.38	.38
71	.31	.32	.33	.34	.35	.35	.36	.37	.37	.38	.38	.39	.39	.40	.40	.40	.40	.40	.40	.40	.41
72	.34	.35	.36	.37	.38	.39	.40	.41	.42	.42	.42	.43	.43	.43	.44	.44	.44	.44	.44	.44	.44
73	.37	.38	.40	.41	.42	.43	.44	.44	.45	.46	.46	.46	.46	.47	.47	.47	.47	.47	.47	.47	.47
74	.40	.42	.43	.45	.46	.47	.48	.48	.49	.50	.50	.50	.51	.51	.51	.51	.51	.52	.52	.52	.52
75	.44	.46	.47	.49	.50	.51	.52	.52	.53	.54	.54	.54	.55	.55	.56	.56	.56	.57	.57	.58	.58
76	.47	.48	.50	.52	.53	.54	.55	.55	.56	.57	.57	.58	.58	.59	.60	.60	.61	.61	.61	.62	.62
77	.50	.52	.54	.56	.57	.58	.59	.60	.60	.61	.61	.62	.62	.63	.63	.63	.64	.64	.64	.65	.65
78	.53	.54	.56	.58	.60	.61	.62	.62	.63	.64	.64	.65	.65	.66	.66	.66	.67	.67	.68	.68	.68
79	.57	.58	.60	.62	.61	.65	.68	.66	.67	.68	.68	.69	.69	.70	.70	.70	.71	.71	.71	.72	.72
80	.60	.62	.64	.66	.68	.69	.70	.70	.71	.72	.72	.72	.73	.73	.74	.74	.75	.75	.75	.76	.76

To be subtracted from the indicated degree. (temperatures 50–59)

To be added to the indicated degree. (temperatures 61–80)

TABLE I.—*Correction to be applied to the reading of Balling's saccharometer for different temperatures*—Continued.

Degree Balling.

To be added to the indicated degree.

Temp. °F.	1°	2°	3°	4°	5°	6°	7°	8°	9°	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°
81	.63	.65	.67	.69	.71	.72	.73	.73	.74	.75	.75	.76	.76	.77	.78	.78	.79	.79	.79	.79	.79
82	.67	.69	.71	.73	.76	.76	.77	.77	.78	.78	.79	.80	.80	.81	.82	.82	.83	.83	.84	.84	.84
83	.72	.74	.76	.78	.81	.81	.81	.82	.82	.82	.83	.84	.85	.86	.87	.87	.87	.87	.87	.87	.88
84	.77	.79	.81	.83	.86	.86	.86	.86	.87	.87	.88	.89	.90	.91	.92	.92	.93	.93	.93	.93	.94
85	.83	.85	.87	.89	.91	.91	.92	.92	.93	.93	.94	.95	.96	.97	.98	.98	.98	.99	.99	.99	.99
86	.88	.90	.92	.94	.96	.96	.97	.98	.98	.99	1.00	1.01	1.02	1.02	1.03	1.03	1.04	1.04	1.04	1.05	1.05
87	.94	.96	.98	.99	1.01	1.02	1.03	1.03	1.04	1.05	1.05	1.06	1.07	1.07	1.08	1.08	1.09	1.10	1.10	1.11	1.11
88	.99	1.01	1.03	1.04	1.06	1.07	1.08	1.09	1.10	1.11	1.11	1.12	1.12	1.13	1.13	1.14	1.15	1.15	1.16	1.17	1.17
89	1.05	1.07	1.09	1.10	1.11	1.12	1.13	1.14	1.15	1.16	1.16	1.17	1.17	1.18	1.18	1.19	1.21	1.21	1.22	1.23	1.23
90	1.11	1.13	1.15	1.16	1.17	1.18	1.19	1.20	1.21	1.22	1.22	1.23	1.23	1.24	1.24	1.25	1.27	1.28	1.29	1.30	1.30
91	1.15	1.17	1.19	1.20	1.21	1.22	1.23	1.24	1.26	1.27	1.27	1.27	1.28	1.28	1.29	1.30	1.32	1.33	1.34	1.35	1.36
92	1.20	1.22	1.24	1.25	1.26	1.27	1.28	1.30	1.31	1.32	1.32	1.33	1.33	1.34	1.34	1.35	1.37	1.38	1.39	1.40	1.41
93	1.24	1.26	1.28	1.29	1.31	1.32	1.33	1.34	1.36	1.37	1.37	1.38	1.39	1.39	1.40	1.40	1.42	1.43	1.44	1.44	1.45
94	1.29	1.31	1.33	1.35	1.36	1.37	1.39	1.40	1.41	1.42	1.42	1.43	1.44	1.44	1.45	1.45	1.47	1.47	1.48	1.49	1.50
95	1.33	1.35	1.37	1.39	1.41	1.42	1.44	1.45	1.46	1.47	1.47	1.48	1.49	1.49	1.50	1.50	1.51	1.52	1.53	1.54	1.54
96	1.38	1.40	1.42	1.44	1.46	1.47	1.49	1.50	1.51	1.52	1.52	1.53	1.54	1.54	1.55	1.55	1.56	1.57	1.57	1.58	1.58
97	1.42	1.44	1.46	1.48	1.51	1.52	1.54	1.55	1.56	1.57	1.57	1.58	1.59	1.59	1.60	1.60	1.62	1.62	1.63	1.63	1.64
98	1.47	1.49	1.51	1.54	1.56	1.57	1.58	1.59	1.60	1.62	1.63	1.63	1.64	1.64	1.65	1.65	1.66	1.67	1.67	1.68	1.68
99	1.51	1.53	1.55	1.58	1.61	1.62	1.63	1.64	1.66	1.67	1.68	1.68	1.69	1.69	1.71	1.71	1.73	1.73	1.73	1.73	1.74
100	1.55	1.57	1.59	1.62	1.66	1.67	1.68	1.69	1.72	1.72	1.73	1.73	1.74	1.76	1.77	1.77	1.78	1.79	1.79	1.79	1.80

N. B.—The correction for indications between 21° and 30° Balling and 50° and 70° F. temperature may be made by adding 0.005 to each correction given in the above Table, column 21°, for each increase in degree Balling. For instance, 25° Balling 70° F., correction to be added to column 21° and 70°. 0.02 + 0.38 = 0.40.

TABLE II.—*Percentage of alcohol by weight and by volume.*

[Recalculated from Gilpin's, Drinkwater's, Squibb's, etc., determination, by EDGAR RICHARDS.]

Specific gravity at 60°/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60°/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60°/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at 60°/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
1.00000	0.00	0.00	.99629	2.50	1.99	.99281	5.00	4.00	.98959	7.50	6.02
0.99992	.05	.04	622	.55	2.03	274	.05	.04	953	.55	.06
984	.10	.08	615	.60	.07	268	.10	.08	947	.60	.10
976	.15	.12	607	.65	.11	261	.15	.12	940	.65	.14
968	.20	.16	600	.70	.15	255	.20	.16	934	.70	.18
961	.25	.20	593	.75	.19	248	.25	.20	928	.75	.22
953	.30	.24	586	.80	.23	241	.30	.24	922	.80	.26
945	.35	.28	579	.85	.27	235	.35	.28	916	.85	.30
937	.40	.32	571	.90	.31	228	.40	.32	909	.90	.34
930	.45	.36	564	.95	.35	222	.45	.36	903	.95	.38
.99923	0.50	0.40	.99557	3.00	2.39	.99215	5.50	4.40	.98897	8.00	6.42
915	.55	.44	550	.05	.43	208	.55	.44	891	.05	.46
907	.60	.48	543	.10	.47	202	.60	.48	885	.10	.50
900	.65	.52	336	.15	.51	195	.65	.52	879	.15	.54
892	.70	.56	529	.20	.55	189	.70	.56	873	.20	.58
884	.75	.60	522	.25	.59	182	.75	.60	867	.25	.62
877	.70	.64	515	.30	.64	175	.80	.64	861	.30	.67
869	.85	.67	508	.35	.68	169	.85	.68	855	.35	.71
861	.90	.71	501	.40	.72	162	.90	.72	849	.40	.75
854	.95	.75	494	.45	.76	156	.95	.76	843	.45	.79
.99849	1.00	0.79	.99487	3.50	2.80	.99149	6.00	4.80	.98837	8.50	6.83
842	.05	.83	480	.55	.84	143	.05	.84	831	.55	.87
834	.10	.87	473	.60	.88	136	.10	.88	825	.60	.91
827	.15	.91	466	.65	.92	130	.15	.92	819	.65	.95
819	.20	.95	459	.70	.96	123	.20	.96	813	.70	.99
813	.25	.99	452	.75	3.00	117	.25	5.00	807	.75	7.03
805	.30	1.03	445	.80	.04	111	·30	.05	801	.80	.07
797	.35	.07	438	.85	.08	104	.35	.09	795	.85	.11
790	.40	.11	431	.90	.12	098	.40	.13	789	.90	.15
782	.45	.15	424	.95	.16	091	.45	.17	783	.95	.19
.99775	1.50	1.19	.99417	4.00	3.20	.99085	6.50	5.21	.98777	9.00	7.23
768	.55	.23	410	.05	.24	079	.55	.25	771	.05	.27
760	.60	.27	403	.10	.28	072	.60	.29	765	.10	.31
753	.65	.31	397	.15	.32	066	.65	.33	759	.15	.35
745	.70	.35	390	.20	.36	059	.70	.37	754	.20	.39
738	.75	.39	383	.25	.40	053	.75	.41	748	.25	.43
731	.80	.43	376	.30	.44	047	.80	.45	742	.30	.48
723	.85	.47	369	.35	.48	040	.85	.49	736	.35	.52
716	.90	.51	363	.40	.52	034	.90	.53	730	.40	.56
708	.95	.55	356	.45	.56	027	.95	.57	724	.45	.60
.99701	2.00	1.59	.99349	4.50	3.60	.99021	7.00	5.61	.98719	9.50	7.64
694	.05	.63	342	.55	.64	015	.05	.65	713	.55	.68
687	.10	.67	335	.60	.68	009	.10	.69	707	.60	.72
679	.15	.71	329	.65	.72	002	.15	.73	701	.65	.76
672	.20	.75	322	.70	.76	.98996	.20	.77	695	.70	.80
665	.25	.79	315	.75	.80	990	.25	.81	689	.75	.84
658	.30	.83	308	.80	.84	984	.30	.86	683	.80	.88
651	.35	.87	301	.85	.88	978	.35	.90	678	.85	.92
643	.30	.91	295	.90	.92	971	.40	.94	672	.90	.96
636	.45	.95	288	.95	.96	965	.45	.98	606	.95	8.00

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.98660	10.00	8.04	.98381	12.50	10.08	.98114	15.00	12.13	.97859	17.50	14.19
654	.05	.08	375	.55	.12	108	.05	.17	853	.55	.23
649	.10	.12	370	.60	.16	104	.10	.21	848	.60	.27
643	.15	.16	364	.65	.20	099	.15	.25	843	.65	.31
637	.20	.20	359	.70	.24	093	.20	.29	838	.70	.35
632	.25	.24	353	.75	.28	088	.25	.33	833	.75	.40
626	.30	.29	348	.80	.33	083	.30	.38	828	.80	.44
620	.35	.33	342	.85	.37	078	.35	.42	823	.85	.48
614	.40	.37	337	.90	.41	073	.40	.46	818	.90	.52
609	.45	.41	331	.95	.45	068	.45	.50	813	.95	.56
.98603	10.50	8.45	.98326	13.00	10.49	.98063	15.50	12.54	.97808	18.00	14.60
597	.55	.49	321	.05	.53	057	.55	.58	803	.05	.64
592	.60	.53	315	.10	.57	052	.60	.62	798	.10	.68
586	.65	.57	310	.15	.61	047	.65	.66	793	.15	.73
580	.70	.61	305	.20	.65	042	.70	.70	788	.20	.77
575	.75	.65	299	.25	.69	037	.75	.75	783	.25	.81
569	.80	.70	294	.30	.74	032	.80	.79	778	.30	.85
563	.85	.74	289	.35	.78	026	.85	.83	773	.35	.89
557	.90	.78	283	.40	.82	021	.90	.87	768	.40	.94
552	.95	.82	278	.45	.86	016	.95	.91	763	.45	.98
98546	11.00	8.86	.98273	13.50	10.90	.98011	16.00	12.95	.97758	18.50	15.02
540	.05	.90	267	.55	.94	005	.05	.99	753	.55	.06
535	.10	.94	262	.60	.98	001	.10	13.03	748	.60	.10
529	.15	.98	256	.65	11.02	.97996	.15	.08	743	.65	.14
524	.20	9.02	251	.70	.06	991	.20	.12	738	.70	.18
518	.25	.07	246	.75	.11	986	.25	.16	733	.75	.22
513	.30	.11	240	.80	.15	980	.30	.20	728	.80	.27
507	.35	.15	235	.85	.19	975	.35	.24	723	.85	.31
502	.40	.19	230	.90	.23	970	.40	.29	718	.90	.35
496	.45	.23	224	.95	.27	965	.45	.33	713	.95	.39
.98491	11.50	9.27	98.219	14.00	11.31	.97960	16.50	13.37	.97708	19.00	15.43
485	.55	.31	214	.05	.35	955	.55	.41	703	.05	.47
479	.60	.35	209	.10	.39	950	.60	.45	698	.10	.51
474	.65	.39	203	.15	.43	945	.65	.49	693	.15	.55
468	.70	.43	198	.20	.47	940	.70	.53	688	.20	.59
463	.75	.47	193	.25	.52	935	.75	.57	683	.25	.63
457	.80	.51	188	.30	.56	929	.80	.62	678	.30	.68
452	.85	.55	182	.35	.60	924	.85	.66	673	.35	.72
446	.90	.59	177	.40	.64	919	.90	.70	668	.40	.76
441	.95	.63	172	.45	.68	914	.95	.74	663	.45	80
.98435	12.00	9.67	.98167	14.50	11.72	.97909	17.00	13.78	.97658	19.50	15.84
430	.05	.71	161	.55	.76	904	.05	.82	653	.55	.88
424	.10	.75	156	.60	.80	899	.10	.86	648	.60	.93
419	.15	.79	151	.65	.84	894	.15	.90	643	.65	.97
413	.20	.83	146	.70	.88	889	.20	.94	638	.70	16.01
408	.25	.87	140	.75	.93	884	.25	.98	633	.75	.05
402	.30	.92	135	.80	.97	879	.30	14.03	628	.80	.09
397	.35	.96	130	.85	12.05	874	.35	.07	623	.85	.14
391	.40	10.00	125	.90	.05	869	.40	.11	618	.90	.18
386	.45	.04	119	.95	.09	864	.45	.15	613	.95	.22

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at ⁶⁰⁄₆₀° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at ⁶⁰⁄₆₀° F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at ⁶⁰⁄₆₀° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at ⁶⁰⁄₆₀° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.97608	20.00	16.26	.97355	22.50	18.34	.97097	25.00	20.43	.96828	27.50	22.54
603	.05	.30	350	.55	.38	092	.05	.47	822	.55	.58
598	.10	.34	345	.60	.42	086	.10	.51	816	.60	.62
593	.15	.38	340	.65	.47	081	.15	.56	811	.65	.67
588	.20	.42	335	.70	.51	076	.20	.60	805	.70	.71
583	.25	.46	330	.75	.55	071	.25	.64	800	.75	.75
578	.30	.51	324	.80	.59	065	.30	.68	794	.80	.79
573	.35	.58	319	.85	.63	060	.35	.72	789	.85	.83
568	.40	.59	314	.90	.68	055	.40	.77	783	.90	.88
563	.45	.63	309	.95	.72	049	.45	.81	778	.95	.92
.97558	20.50	16.67	.97304	23.00	18.76	.97044	25.50	20.85	.96772	28.00	22.96
552	.55	.71	299	.05	.80	039	.55	.89	766	.05	23.00
547	.60	.75	294	.10	.84	033	.60	.93	761	.10	.04
542	.65	.80	289	.15	.88	028	.65	.98	755	.15	.09
537	.70	.84	283	.20	.92	023	.70	21.02	749	.20	.13
532	.75	.88	278	.25	.96	018	.75	.06	744	.25	.17
527	.80	.92	273	.30	19.01	012	.80	.10	738	.30	.21
522	.85	.96	268	.35	.05	007	.85	.14	732	.35	.25
517	.90	17.01	263	.40	.09	001	.90	.19	726	.40	.30
512	.95	.05	258	.45	.13	.96996	.95	.23	721	.45	.34
.97507	21.00	17.09	.97253	23.50	19.17	.96991	26.00	21.27	.96715	28.50	23.38
502	.05	.13	247	.55	.21	986	.05	.31	709	.55	.42
497	.10	.17	242	.60	.25	980	.10	.35	704	.60	.47
492	.15	.22	237	.65	.30	975	.15	.40	698	.65	.51
487	.20	.26	232	.70	.34	969	.20	.44	692	.70	.55
482	.25	.30	227	.75	.38	964	.25	.48	687	.75	.60
477	.30	.34	222	.80	.42	959	.30	.52	681	.80	.64
472	.35	.38	216	.85	.46	953	.35	.56	675	.85	.68
467	.40	.43	211	.90	.51	949	.40	.61	669	.90	.72
562	.45	.47	206	.95	.55	942	.45	.65	664	.95	.77
.97457	21.50	17.51	.97201	24.00	19.59	.96937	26.50	21.69	.96658	29.00	23.81
451	.55	.55	196	.05	.63	932	.55	.73	652	.05	.85
446	.60	.59	191	.10	.67	926	.60	.77	646	.10	.89
441	.65	.63	185	.15	.72	921	.65	.82	640	.15	.94
436	.70	.67	180	.20	.76	915	.70	.86	635	.20	.98
431	.75	.71	175	.25	.80	910	.75	.90	629	.25	24.02
426	.80	.76	170	.30	.84	905	.80	.94	623	.30	.06
421	.85	.80	165	.35	.88	899	.85	.98	617	.35	.10
416	.90	.84	159	.40	.93	894	.90	22.03	611	.40	.15
411	.95	.88	154	.45	.97	888	.95	.07	605	.45	.19
.97406	22.00	17.92	.97149	24.50	20.01	.96883	27.00	22.11	.96600	29.50	24.23
401	.05	.96	144	.55	.05	877	.05	.15	594	.55	.27
396	.10	18.00	139	.60	.09	872	.10	.20	587	.60	.32
391	.15	.05	133	.65	.14	866	.15	.24	582	.65	.36
386	.20	.03	128	.70	.18	861	.20	.28	576	.70	.40
381	.25	.13	123	.75	.22	855	.25	.33	570	.75	.45
375	.30	.17	118	.80	.26	850	.30	.37	564	.80	.49
370	.35	.21	113	.85	.30	844	.35	.41	559	.85	.53
365	.40	.26	107	.90	.35	839	.40	.45	553	.90	.57
360	.45	30	102	.95	.39	833	.45	.50	547	.95	.62

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.96541	30.00	24.66	.96235	32.50	26.80	.95910	35.00	28.96	.95560	37.50	31.14
535	.05	.70	229	.55	.84	903	.05	29.00	552	.55	.18
529	.10	.74	222	.60	.89	896	.10	.05	545	.60	.23
523	.15	.79	216	.65	.93	889	.15	.09	538	.65	.27
517	.20	.83	210	.70	.97	883	.20	.13	531	.70	.32
511	.25	.87	204	.75	27.02	876	.25	.18	523	.75	.36
505	.30	.91	197	.80	.06	869	.30	.22	516	.80	.40
499	.35	.95	191	.85	.10	862	.35	.26	509	.85	.45
493	.40	25.00	185	.90	.14	855	.40	.30	502	.90	.49
487	.45	.04	178	.95	.19	848	.45	.35	494	.95	.54
.96181	30.50	25.08	.96172	33.00	27.23	.95842	35.50	29.39	.95487	38.00	31.58
475	.55	.12	166	.05	.27	835	.55	.43	480	.05	.63
469	.60	.17	159	.10	.32	828	.60	.48	472	.10	.67
463	.65	.21	153	.15	.36	821	.65	.52	465	.15	.72
457	.70	.25	146	.20	.40	814	.70	.57	457	.20	.76
451	.75	.30	140	.25	.45	807	.75	.61	450	.25	.81
445	.80	.34	133	.30	.49	800	.80	.65	442	.30	.85
439	.85	.38	127	.35	.53	794	85	.70	435	.35	.90
433	.90	.42	120	.40	.57	787	.90	.74	427	.40	.94
427	.95	.47	114	.45	.62	780	.95	.79	420	.45	.99
06421	31.00	25.51	.96108	33.50	27.66	.95773	36.00	29.83	.95413	8.50	32.03
415	.05	.55	101	.55	.70	766	.05	.87	405	.55	.07
409	.10	.60	095	.60	.75	759	.10	.92	398	.60	.12
403	.15	.64	088	.65	.79	752	.15	.96	390	.65	.16
396	.20	.68	082	.70	.83	745	.20	30.00	383	.70	.20
390	.25	.73	075	.75	.88	738	.25	.05	375	.75	.25
381	.30	.77	069	.80	.92	731	.30	.09	368	.80	.29
378	.35	.81	062	.85	.96	724	.35	.13	360	.85	33
372	.40	,85	056	.90	28.00	717	.40	.17	353	.90	.37
366	.45	.90	049	.95	.05	710	.45	.22	345	.95	.42
.96369	31.50	25.94	.96043	34.00	28.09	.95703	36.50	30.26	.95338	39.00	32.46
353	.55	.98	036	.05	.13	695	.55	.30	330	.05	.50
347	.60	26.03	030	.10	.18	688	.60	.35	323	.10	.55
341	.65	.07	023	.15	.22	681	.65	.39	315	.15	.59
335	.70	.11	016	.20	.26	674	.70	.44	307	.20	.64
329	.75	.16	010	.25	.31	667	.75	.48	300	.25	.68
323	.80	.20	003	.30	.35	660	.80	.52	292	.30	.72
316	.85	.24	.95996	.35	.39	653	.85	.57	284	.35	.77
310	.90	.28	990	.40	.43	646	.90	.61	277	.40	.81
304	.95	.33	983	.45	.48	639	.95	.66	269	.45	.86
.96298	32.00	26.37	.95977	34.50	28.52	.95632	37.00	30.70	.95262	89.50	32.90
292	.05	.41	970	.55	.56	625	.05	.74	254	.55	.95
285	.10	.46	963	.60	.61	618	.10	.79	246	.60	.99
279	.15	.50	957	.65	.65	610	.15	.83	239	.65	33.04
273	.20	.54	950	.70	.70	603	.20	.88	231	.70	.08
267	.25	.59	943	.75	.74	596	.25	.92	223	.75	.13
260	.30	.63	937	.80	.78	589	.30	.96	216	.80	.17
254	.35	.67	930	.85	.83	581	.35	31.01	208	.85	.22
248	.40	.71	923	.90	.87	574	.40	.05	200	.90	.27
241	.45	.76	917	.95	.92	567	.45	.10	193	.95	.31

TABLE II.—*Percentage of alcohol by weight and by volume*—Continued.

Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60/60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.95185	40.00	33.35	.94786	42.50	35.58	.94364	45.00	37.84	.93916	47.50	40.13
177	.05	.39	778	.55	.63	355	.05	.89	906	.55	.18
169	.10	.44	770	.60	.67	346	.10	.93	898	.60	.22
161	.15	.48	761	.65	.72	338	.15	.98	888	.65	.27
154	.20	.53	753	.70	.76	329	.20	38.02	879	.70	.32
146	.25	.57	745	.75	.81	320	.25	.07	870	.75	.37
138	.30	.61	737	.80	.85	311	.30	.12	861	.80	.41
130	.35	.66	729	.85	.90	302	.35	.16	852	.85	.46
122	.40	.70	720	.90	.94	294	.40	.21	842	.90	.51
114	.45	.75	712	.95	.99	285	.45	.25	833	.95	.55
.95107	40.50	33.79	.94704	43.00	36.03	.94276	45.50	38.30	.93824	48.00	40.60
099	.55	.84	696	.05	.08	267	.55	.35	815	.05	.65
091	.60	.88	687	.10	.12	258	.60	.39	805	.10	.69
083	.65	.93	679	.15	.17	250	.65	.44	796	.15	.74
075	.70	.97	670	.20	.21	241	.70	.48	786	.20	.78
067	.75	34.02	662	.25	.26	232	.75	.53	777	.25	.83
059	.80	.06	654	.30	.30	223	.80	.57	768	.30	.88
052	.85	.11	645	.35	.35	214	.85	.62	758	.35	.92
044	.90	.15	637	.40	.39	206	.90	.66	749	.40	.97
036	.95	.20	628	.45	.44	197	.95	.71	739	.45	41.01
.95028	41.00	34.24	.94620	43.50	36.48	.94188	46.00	38.75	.93730	48.50	41.06
020	.05	.28	612	.55	.53	179	.05	.80	721	.55	.11
012	.10	.33	603	.60	.57	170	.10	.84	711	.60	.15
004	.15	.37	595	.65	.62	161	.15	.89	702	.65	.20
.94996	.20	.42	586	.70	.66	152	.20	.93	692	.70	.24
988	.25	.46	578	.75	.71	143	.25	.98	683	.75	.29
980	.30	.50	570	.80	.75	134	.30	39.03	679	.80	.34
972	.35	.55	561	.85	.80	125	.35	.07	664	.85	.38
964	.40	.59	553	.90	.84	116	.40	.12	655	.90	.43
956	.45	.64	544	.95	.89	107	.45	.16	645	.95	.47
.94948	41.50	34.68	.94536	44.00	36.93	.94098	46.50	39.21	.93636	49.00	41.52
940	.55	.73	527	.05	.98	089	.55	.26	626	.05	.57
932	.60	.77	519	.10	37.02	080	.60	.30	617	.10	.61
924	.65	.82	510	.15	.07	071	.65	.35	607	.15	.66
916	.70	.86	502	.20	.11	062	.70	.39	598	.20	.71
908	.75	.91	493	.25	.16	053	.75	.44	588	.25	.76
900	.80	.95	484	.30	.21	044	.80	.49	578	.30	.80
892	.85	35.00	476	.35	.25	035	.85	.53	569	.35	.85
884	.90	.04	467	.40	.30	026	.90	.58	559	.40	.90
876	.95	.09	459	.45	.34	017	.95	.62	550	.45	.94
.94868	42.00	35.13	.94450	44.50	37.39	.94008	47.00	39.67	.93540	49.50	41.99
860	.05	.18	441	.55	.44	.93999	.05	.72	530	.55	42.04
852	.10	.22	433	.60	.48	990	.10	.76	521	.60	.08
843	.15	.27	424	.65	.53	980	.15	.81	511	.65	.13
835	.20	.31	416	.70	.57	671	.20	.85	502	.70	.18
827	.25	.36	407	.75	.62	962	.25	.90	492	.75	.23
819	.30	.40	398	.80	.66	953	.30	.95	482	.80	.27
811	.35	.45	390	.85	.71	944	.35	.99	473	.85	.32
802	.40	.49	381	.90	.76	934	.40	40.04	463	.90	.37
794	.45	.54	373	.95	.80	925	.45	.08	454	.95	.41

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TABLE OF ATOMIC WEIGHTS.

[Issued December 6, 1890.]

Revised by F. W. CLARKE, *Chief Chemist of the United States Geological Survey.*

[This table represents the latest and most trustworthy results, reduced to a uniform basis of comparison, with oxygen=16 as starting point of the system. No decimal places representing large uncertainties are used. When values vary, with equal probability on both sides, so far as our present knowledge goes, as in the case of cadmium (111.8 and 112.2), the mean value is given in the table.]

Name.	Symbol.	Atomic weight.	Name.	Symbol.	Atomic weight.
Aluminum	Al	27	Molybdenum	Mo	96
Antimony	Sb	120	Nickel	Ni	58.7
Arsenic	As	75	Nitrogen	N	14.03
Barium	Ba	137	Osmium	Os	190.8
Bismuth	Bi	208.9	Oxygen[4]	O	16
Boron	B	11	Palladium	Pd	106.6
Bromin	Br	79.95	Phosphorus	P	31
Cadmium	Cd	112	Platinum	Pt	195
Cesium	Cs	132.9	Potassium	K	39.11
Calcium	Ca	40	Rhodium	Rh	103
Carbon	C	12	Rubidium	Rb	85.5
Cerium	Ce	140.2	Ruthenium	Ru	101.6
Chlorin	Cl	35.45	Samarium	Sm	150
Chromium	Cr	52.1	Scandium	Sc	44
Cobalt	Co	59	Selenium	Se	79
Columbium[1]	Cb	94	Silicon	Si	28.4
Copper	Cu	63.6	Silver	Ag	107.92
Didymium[2]	Di	142.3	Sodium	Na	23.05
Erbium	Er	166.3	Strontium	Sr	87.6
Fluorin	F	19	Sulphur	S	32.06
Gallium	Ga	69	Tantalum	Ta	182.6
Germanium	Ge	72.3	Tellurium	Te	125
Glucinum[3]	Gl	9	Terbium	Tb	159.5
Gold	Au	197.3	Thallium	Tl	204.18
Hydrogen	H	1.007	Thorium	Th	232.6
Indium	In	113.7	Tin	Sn	119
Iodin	I	126.85	Titanium	Ti	48
Iridium	Ir	193.1	Tungsten	W	184
Iron	Fe	56	Uranium	U	239.6
Lanthanum	La	138.2	Vanadium	V	51.4
Lead	Pb	206.95	Ytterbium	Yb	173
Lithium	Li	7.02	Yttrium	Yt	89.1
Magnesium	Mg	24.3	Zinc	Zn	65.3
Manganese	Mn	55	Zirconium	Zr	90.6
Mercury	Hg	200			

[1] Has priority over Niobium.
[2] Now split into Neo- and Praseo-Didymium.
[3] Has priority over Beryllium.
[4] Standard or basis of the system.

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A.

C.

D.